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 KLEINFELDER

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# SOUTHERN CALIFORNIA CHEMICAL

A DIVISION OF CP CHEMICALS, INC.

8851 DICE ROAD • SANTA FE SPRINGS, CALIFORNIA 90670-0118

June 9, 1988

Mr. Hank H. Yacoub  
Supervising Water Resources  
Control Engineer  
California Regional Water Quality  
Control Board  
Los Angeles Region  
107 South Broadway, Room 4027  
Los Angeles, California 90012-4596


Re: Quality Assurance Project Plan  
for Groundwater Sampling Program

Dear Mr. Yacoub:

Enclosed is a copy of the above referenced plan for Southern California Chemical as requested at the CME review meeting on February 4, 1988.

We would appreciate receiving written comments from all concerned as quickly as possible.

Very truly yours,

  
Gregor Otterbach  
Vice President

GO:lat  
Enclosure

cc with enclosure:  
Mr. Jim Breitlow, U.S. EPA, San Francisco  
Mr. Mark Vest, DHS, Los Angeles  
Mr. Brian Lewis, DHS, Sacramento  
Ms. Nancy Ball, DHS, Sacramento  
Mr. R. E. Torrance  
Mr. J. S. Leo  
Mr. J. L. Benjamin  
Mr. M. Giorgetta

Certified Mail -  
Return Receipt Requested

SOUTHERN CALIFORNIA CHEMICAL COMPANY  
SAMPLING PROGRAM

Prepared For

Southern California Chemical Company  
8851 Dice Road  
Santa Fe Springs, California

Prepared By

Kleinfelder  
17100 Pioneer Boulevard  
Suite 350  
Artesia, California 90701

May 1988

1 TITLE AND SIGNATURE PAGE


Quality Assurance Project Plan For  
Southern California Chemical Company  
Groundwater Sampling Program

(50-1014-03)

Prepared By

Kleinfelder  
17100 Pioneer Boulevard  
Suite 350  
Artesia, California 90701

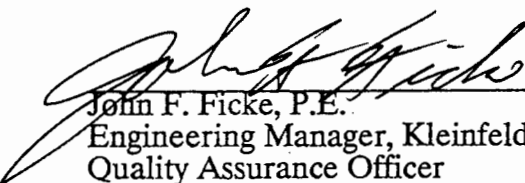
APPROVALS:

  
Greg Otterbach  
Project Director,  
Southern California Chemical Company

6/6/88  
Date

  
Kenneth Durand, Project Hydrogeologist  
Project Manager, Kleinfelder

5/30/88  
Date

  
John F. Ficke, P.E.  
Engineering Manager, Kleinfelder  
Quality Assurance Officer

May 31, 1988  
Date

## 2 TABLE OF CONTENTS

SECTION	PAGE	REVISION	DATE
1.0 TITLE PAGE	1	0	5/27/88
2.0 TABLE OF CONTENTS	2	0	5/27/88
3.0 PROJECT DESCRIPTION	4	0	5/27/88
4.0 PROJECT ORGANIZATION	6	0	5/27/88
5.0 QUALITY ASSURANCE	8	0	5/27/88
6.0 SAMPLING PROCEDURES	9	0	5/27/88
7.0 SAMPLE CUSTODY PROCEDURES	13	0	5/27/88
8.0 CALIBRATION PROCEDURES AND FREQUENCY	15	0	5/27/88
9.0 ANALYTICAL PROCEDURES	16	0	5/27/88
10.0 DATA VALIDATION PROCEDURES	17	0	5/27/88
11.0 INTERNAL QUALITY CONTROL CHECKS AND FREQUENCY	18	0	5/27/88
12.0 PERFORMANCE AND SYSTEMS AUDITS	20	0	5/27/88
13.0 PREVENTATIVE MAINTENANCE PROCEDURES	22	0	5/27/88
14.0 DATA ASSESSMENT PROCEDURES	23	0	5/27/88
15.0 CORRECTIVE ACTION	25	0	5/27/88
16.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT	26	0	5/27/88

## TABLE OF CONTENTS (continued)

### TABLES

- 1 Monitoring Well Data
- 2 Specific Duties and Responsibilities of the Project Manager
- 3 Specific Duties and Responsibilities of the Quality Assurance Officer
- 4 Sampling and Preservation Requirements
- 5 Sampling Frequency

### FIGURES

- 1 Monitoring Well Location Map
- 2 Project Organization Chart
- 3 Chain of Custody Forms

### LIST OF APPENDICES

- A Laboratory Quality Assurance Plan, Chemical Research Laboratories
- B Laboratory Quality Assurance Plan, Brown and Caldwell Laboratories

### 3 PROJECT DESCRIPTION

#### 3.1 INTRODUCTION

The purpose of this project is to characterize the groundwater quality beneath the Southern California Chemical Company (SCC) facility at 8851 Dice Road in Santa Fe Springs, California.

SCC has been located at its present address for over 28 years. The facility manufactures inorganic chemicals for plating, printed circuitry, water treatment, and agriculture uses. Chemicals used on site include copper sulfate, copper chloride, zinc sulfate, nickel, and ferric chloride.

Significant characterization activities have already been performed at this site. A program of routine sampling and analysis of the on-site groundwater has been in effect since February 1985. In addition, numerous on-site soil samples have been collected to characterize the on-site distribution of the chemicals of concern.

Conclusions of the previous studies were:

- o Hexavalent chromium exists at concentrations above federal drinking water standards in the groundwater beneath Pond 1. The source of hexavalent chromium appears to have been due to leakage from an old tank located 60 feet east of Pond 1.
- o Elevated concentrations of organic compounds exist in the groundwater beneath the northern boundary and the center of the facility. The source of the organic chemicals is probably a neighboring facility.

All previous reports are available at the Department of Health Services (DHS), and the California State Regional Water Quality Control Board, Los Angeles Region (RWQCB) offices if a more detailed discussion of the site investigations is desired.

These reports are listed below:

<u>Date</u>	<u>Report</u>
June 1985	Environmental Monitoring Study (Phase I)
October 1985	Hydrogeologic Assessment Pond Number 1
January 1986	Environmental Assessment (Phase II)
March 1986	Environmental Assessment (Phase III)

In addition to the above reports, eight quarterly groundwater sampling reports have been submitted to the agencies.

Groundwater monitoring data generated is used to:

- o Evaluate the three-dimensional distribution of constituents in the groundwater.
- o Evaluate the rate and direction of migration of the chromium and organic chemical plumes.

- o Evaluate the source of the compounds of concern.
- o Evaluate the risk to public health and environment associated with the identified constituents.
- o Evaluate various remedial options judged as necessary to reduce the risk to public health and environment associated with the identified constituents.

Quality assurance procedures must be implemented so that the generated field and laboratory data are of high quality and that all project work is performed in accordance with professional standards. Outlined in this Quality Assurance Project Plan (QAPP) are the procedures to be followed so that the data collected is reliable and of high quality. The main text describes the field and data management quality assurance procedures which will be used to assess the SCC site. Appendix A and Appendix B describe the laboratory quality assurance procedures which will be used by the respective labs.

### 3.2 MONITORING WELL NETWORK

The existing monitoring well network consists of 13 wells, the location of which are shown on Figure 1, Monitoring Well Location Map. Eleven of these wells are 2-inch diameter, the other two are 4-inch wells. All wells are located within the facility boundary.

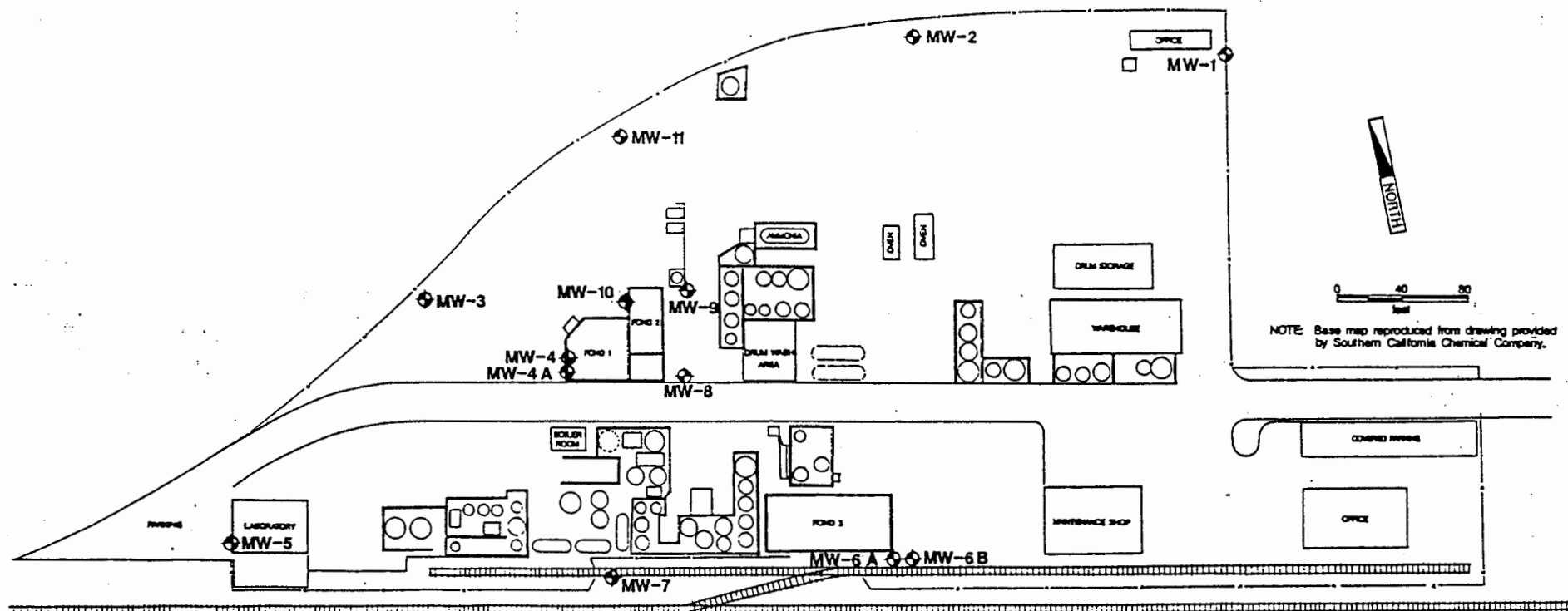
Monitoring wells within the network are screened in three depth zones. Monitoring well MW-6A, which is screened between 10 and 30 feet below ground surface, is above the confining clay zone, which is underlain by the "first water". MW-6A, the only well in this zone, was installed as a result of the detection of a wet zone during drilling. Subsequently the well has remained dry and no samples have been collected.

Eleven monitoring wells are all perforated in the uppermost portion of the first aquifer, approximately 45 to 75 feet below ground surface. Monitoring well MW-4A is perforated 87 to 107 feet below ground surface in the deeper portion of the first aquifer.

The monitoring wells are constructed of either 2 or 4 inch diameters (Table 1) Schedule 40 PVC casing. The screened interval of each well is constructed of 0.020 inch width machine slotted PVC, surrounded by #3 Monterey type sand. Above the sand is a bentonite seal overlain by Volclay grout. The upper few feet are sealed with concrete. Monitoring wells 4A and 5 were constructed with above ground 8 inch diameter locking well protectors. The remaining wells were constructed with flush mounted aluminum water tight well covers set 1½ inches above grade.



Checked by: \_\_\_\_\_ Date: \_\_\_\_\_  
Drawn by: \_\_\_\_\_ Date: \_\_\_\_\_



### EXPLANATION

◆ MONITORING WELL, estimated location

SOUTHERN CALIFORNIA CHEMICAL COMPANY  
Santa Fe Springs, California

MONITORING WELL  
LOCATION MAP



KLEINFELDER

Project Number 50-1014-02

April 1983

FIGURE  
1

**TABLE 1**  
**MONITORING WELL DATA**

Well Number	Well Head Elevation (feet MSL)	Feet Below Ground Surface		Diameter of Well
		Well Depth	Perforated Interval	
MW-1	152.62	62.5	42.5-62.5	2"
MW-2	151.56	74.0	44-74	2"
MW-3	151.62	75.0	45-75	2"
MW-4	149.76	75.0	45-75	2"
MW-4A	152.49	107.0	87-107	4"
MW-5	153.21	75.0	45-75	2"
MW-6A	149.31	30.0	10-30	2"
MW-6B	149.46	77.0	45-77	2"
MW-7	149.27	75.0	45-75	2"
MW-8	149.53	71.0	41-71	2"
MW-9	151.14	77.0	47-77	4"
MW-10	151.60	75.0	45-75	2"
MW-11	152.80	75.5	55-75	2"

NOTE: MSL = Elevations in feet above mean sea level.

## 4 PROJECT ORGANIZATION

The purpose of the QA/QC plan is to provide the guidelines and rules to achieve accuracy, precision, and reliability of the data. The success of the QA/QC program depends on the awareness and cooperation of each individual participating in the project.

Field sampling and data management activities for this project will be performed under the direction of Kleinfelder. The project organization chart (Figure 2) shows the individuals responsible for the quality assurance/quality control objectives and procedures detailed in this plan.

Two people have primary responsibility for assuring and controlling the quality of work for Kleinfelder on this project. They are the Project Manager, and the Quality Assurance/Quality Control (QA/QC) officer. The specific duties and responsibilities of the various project team members as they relate to QA/QC are outlined below. Primary laboratory analyses for the project will be performed by Chemical Research Laboratory. Analyses of duplicate (split) samples will be performed by Brown and Caldwell Laboratory. The respective laboratory Quality Control Directors will be responsible for assuring that all samples analyzed at their labs are analyzed in accordance with approved QA/QC procedures. The SCC Project Director will provide overall oversight of all QA/QC activities to assure that approved procedures are followed.

### 4.1 KLEINFELDER ORGANIZATION

Project Manager. The Project Manager has overall responsibility for technical quality, cost control, personnel management, and scheduling. He is ultimately responsible for the quality of work performed on the project. Mr. Ken Durand serves as the Project Manager for Kleinfelder on this project. His specific responsibilities are given in Table 2.

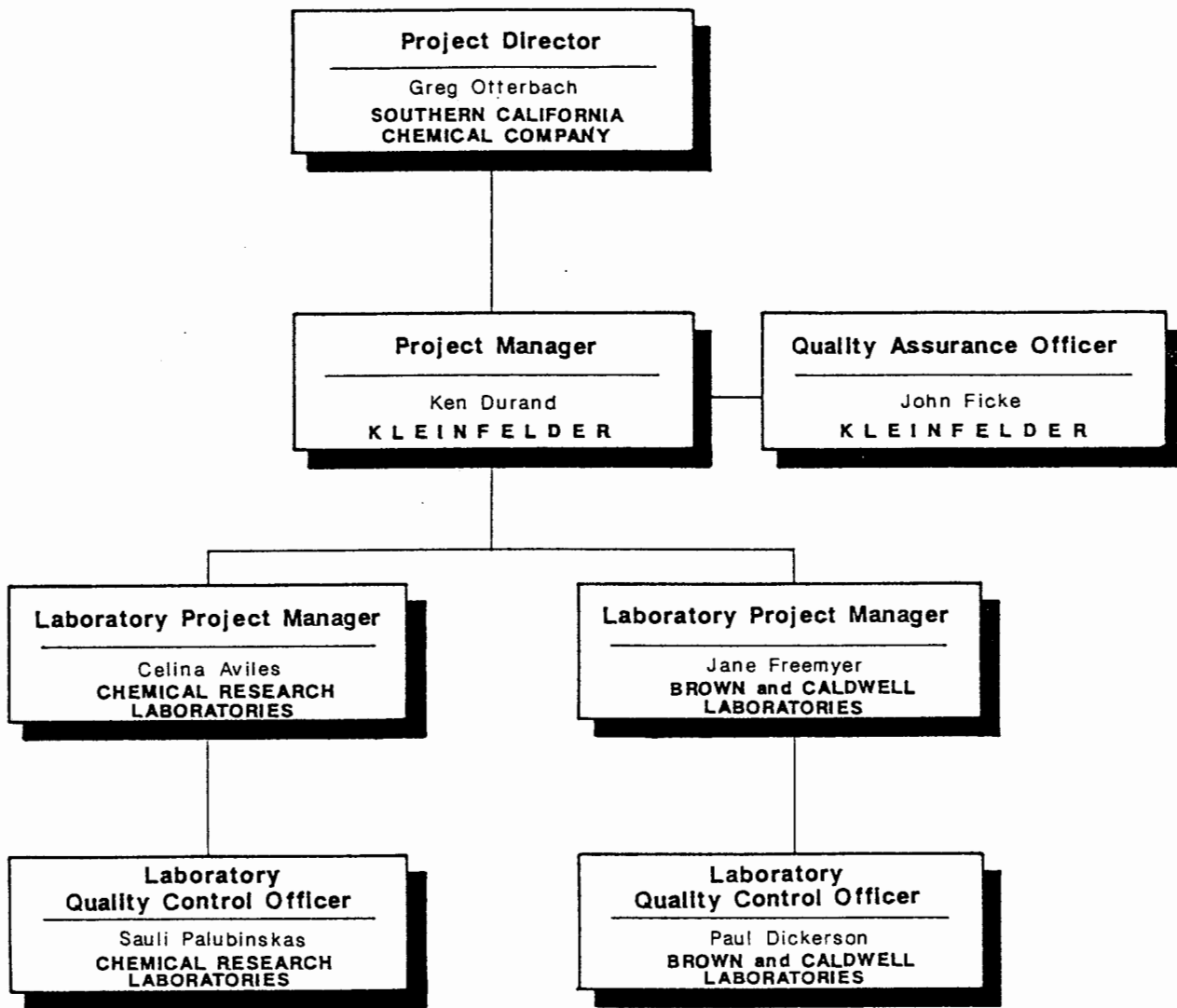
Quality Assurance/Quality Control Officer. The role of the QA/QC officer will be to maintain open lines of communication and inform the project staff of QA/QC protocols. Furthermore, the QA/QC will review analytical products and deliverables for conformance to established limits and agreed-to structures. Mr. John Ficke will serve as the QA/QC Officer for Kleinfelder on this project. His specific responsibilities are given in Table 3.

### 4.2 SOUTHERN CALIFORNIA CHEMICAL COMPANY

Project Director. The SCC Project Director is Mr. Greg Otterbach. He has overall responsibility for oversight of the performance and scheduling of QA/QC activities. He will ensure that the performance of all contractors meets the approved QA/QC procedures. He will maintain a close working relationship with the appropriate regulatory agency staff so that the QA/QC program is effective and continues to meet project objectives.

### 4.3 CHEMICAL RESEARCH LABORATORY

Laboratory Project Manager. Ms. Celina Aviles serves as the Laboratory Project Manager for Chemical Research Laboratory. She has overall project responsibility for the laboratory activities required for the completion of all analyses related to the assessment of the SCC site. She will function as the primary liaison between the laboratory and the project management team. Her specific duties are outlined in Appendix A, Section 4.0.



## PROJECT ORGANIZATION

FIGURE 2

**TABLE 2**  
**SPECIFIC DUTIES AND RESPONSIBILITIES OF THE**  
**PROJECT MANAGER**

- 
- o Responsible for overall technical direction, performance, and scheduling of activities.
  - o Establish and maintain close working relationships with the appropriate RWQCB and DHS staff to implement an effective program.
  - o Participate in task activities where appropriate.
  - o Provide support, guidance and assistance to project managers in planning and organizing technical efforts.
  - o Ensure the performance of subcontractors.
  - o Approve each contract report and deliverable.
  - o Resolve interdisciplinary or technical problems, questions, or priorities to meet project commitments.

TABLE 3

SPECIFIC DUTIES AND RESPONSIBILITIES OF THE  
QUALITY ASSURANCE/QUALITY CONTROL OFFICER

- 
- o Maintains proper lines of communication with the project management team.
  - o Enforce the quality control procedures for reports and deliverables.
  - o Select an impartial peer reviewer for task final reports and deliverables.
  - o Ensure that the formal sign-off procedures are followed and that proper records are kept.
  - o Take any corrective action necessary to preserve quality control of project activities.

Laboratory Quality Control Officer. Ms. Sauli Palubinskas serves as the Laboratory Quality Control Officer for Chemical Research Laboratory. She has project responsibility to ensure that laboratory activities related to the quality assurance of all data are implemented, reviewed, and maintained. In addition, she will implement and manage EPA check programs and round-robin control checks with other outside independent laboratories. Her specific duties are outlined in Appendix A, Section 4.0

#### 4.4 BROWN AND CALDWELL LABORATORY

Laboratory Project Manager. Ms. Jane Freemyer serves as the Laboratory Project Manager for Brown and Caldwell Laboratory. She has overall project responsibility for the laboratory activities required for the completion of all analyses related to the assessment of the SCC site. She will function as the primary liaison between the laboratory and the project management team. Her specific duties are outlined in Appendix B, Section 4.0.

Laboratory Quality Control Officer. Mr. Paul Duerkson serves as the Laboratory Quality Control Officer for Brown and Caldwell. He has project responsibility to ensure that laboratory activities related to the quality assurance of all data are implemented, reviewed, and maintained. In addition, he will implement and manage EPA check programs and round-robin control checks with other outside independent laboratories. His specific duties are outlined in Appendix A, Section 4.0.

## 5 QUALITY ASSURANCE OBJECTIVES

Quality assurance is an integrated program for assuring the reliability of monitoring and measurement data. For groundwater sampling programs, quality assurance means that all samples will be collected, analyzed and reported in accordance with a well defined set of procedures. In general terms, the quality assurance objectives for this project are:

- o Data should be accurate in terms of their agreement with reference or true values.
- o Data should be precise in that there is agreement among individual measurements made under similar conditions.
- o Data should be comparable to prior relevant data for evaluation purposes.
- o Data should be reproducible under similar conditions.
- o Data should be representative of the conditions actually present at the site.
- o Data should be sufficient for appropriate evaluation of the remedial alternatives proposed for final remediation at the site.

Quality assurance objectives are often stated in terms of the precisions, accuracy, and completeness of the data generated. Precision is the degree of agreement among individual measurements made under prescribed conditions. Comparison of samples run in duplicate provides a good indication of precision. Accuracy is the difference between an average measured value and true value, when the latter is known or assumed. Accuracy for some methods is determined by instrument specifications. Comparison of spike concentrations and reported concentrations provides a good mechanism for evaluating accuracy. Completeness is described as the ratio of acceptable laboratory results to the total number of analyses performed. Methods for determining accuracy, precision, and completeness are described in Section 14. Quality assurance objectives pertaining to accuracy and precision of laboratory results for the groundwater sampling programs are summarized in Appendices A and B.



## 6 SAMPLING PROCEDURES

Only groundwater sampling will be carried out on this project. The groundwater sampling procedures are described in the following section for monitoring wells without dedicated pumps and lines. Additional sections may be added to the QAPP in the future to describe soil sampling and off-site domestic well sampling methodologies if required.

### 6.1 MONITORING WELL SAMPLING PROTOCOL

Decontamination, purging, and sampling will be performed by a Kleinfelder environmental technician using the Kleinfelder custom built groundwater sampling vehicle. This vehicle was designed so only teflon and stainless steel components are in contact with the water sample, thereby reducing the potential of water quality distortion.

#### 6.1.1 DECONTAMINATION

The following procedural details describe the method used in decontamination of groundwater sampling equipment prior to sample collection:

- o Exterior surfaces of sampling lines are decontaminated by steam-cleaning during withdrawal from every well.
- o Sample pump is steam-cleaned and rinsed in distilled water.
- o Teflon sampler lines are pressure washed with 5 to 10 gallons of clean, hot water through direct connection to steam-cleaner.
- o Five gallons of distilled water is then pumped through the entire system.
- o Prior to sample collection, a minimum three to five well volumes is purged from the well to permit collection of a representative groundwater sample from the aquifer.

#### 6.1.2 PURGE VOLUME DETERMINATION

The following procedure is followed to determine the appropriate purging volume prior to well sampling.

- o The depth-to-water is measured by a clean, electric water level indicator, which is cleaned with distilled water between wells. Measurement datum is the top of fill ring or top of well protector.
- o Depth to the bottom of the well is measured by a distilled-water rinsed tape and plumb bob. This is compared with the well construction log to determine inconsistencies, i.e., damaged casing, sediment in casing, etc.
- o Water volume is calculated by multiplying total water depth by the inside cross-sectional area of the casing. This figure is one well volume.

#### 6.1.3 WELL PURGING AND SAMPLING

- o Prior to sampling, a minimum of three to five well volumes of water is purged from each well to ensure that water sampled is representative of the groundwater within the formation.
- o Measurements of pH, conductivity, and temperature are taken at frequent intervals during the purge. Stabilization of these values indicates that representative formation water is being removed from the well.

- o In the event that the well is pumped dry, an alternate procedure will be followed. Once a well is pumped dry, the water that enters the well during recovery is, by definition, representative formation water. The well will, therefore, be pumped dry and allowed to recover to 80 percent or more of the original water level. If recovery exceeds two hours, then the sample will be collected as soon as sufficient volume is available.
- o Purge water is pumped directly into barrels onsite until the proper method of disposal is determined.
- o Samples are discharged directly into sampling bottles prepared by the state certified laboratory. The samples are labeled and placed in coolers on ice for transport to the laboratory.
- o Samples are delivered directly to the lab on the same day they collected, whenever practical. If next day delivery is necessary, the samples are kept refrigerated at 4 degrees C overnight in a secure refrigerator and delivered to the laboratory the following morning.
- o Samples are accompanied by a chain-of-custody form which documents the time, date, and responsible person during each step of the transportation process.
- o The Kleinfelder coded sample numbering system allows identification of sample and client to Kleinfelder, while not revealing the client to the laboratory or other interested parties.

Water samples are numbered in the following manner:

W-XX-YY

Where:

W - designates water sample

XX - well number

YY - sequential sample number

For example, W-01-22 indicates a water sample from well number 1. The sample is the 22nd water sample taken at the site.

- o The complete information on the sample label includes:
  - Date and time
  - Client job number (never client name)
  - Sample number
  - Initials of sampler
  - Analysis desired (if known)
  - Preservatives in sample bottle (usually noted by lab).
- o Each sample bottle is given a separate sequential number.

#### 6.1.4 DETECTION OF IMMISCIBLE LAYERS

The following procedure is followed to evaluate the presence of immiscible layers:

- o Prior to the first purging and sampling of each monitoring well. The head space will be analyzed for organic vapors using a photoionization detector.
- o The depth-to-water and depth to product will be determined by use of an interface probe. The difference between the depth to water and depth to product will be the thickness of the immiscible layer.

#### 6.2 SAMPLE CONTAINERS, PRESERVATION, AND HOLDING TIMES

All water samples for this project will be collected, stored, and preserved in accordance with the requirements summarized in Table 4. All containers used for sample storage will be obtained from the laboratory performing the analysis. These containers will be prepared as described in Appendix A and Appendix B.

#### 6.3 SAMPLE COLLECTION AND SEQUENCE

##### 6.3.1 SAMPLE COLLECTION

Samples will be transferred from the pump discharge line directly into the sample containers specifically prepared for each type of chemical analysis (section 6.2).

When collecting the organic chemical samples, the liquid should be introduced into the vials gently to reduce agitation which might drive off volatile compounds. The samples should be placed into the vial without introducing any air bubbles within the vial as it is being filled. Should bubbling occur sample must be poured out and the vial refilled. Each vial should be filled until there is a meniscus over the lip of the vial. The screw-top lid with the septum (Teflon side toward the sample) should then be tightened onto the vial. After tightening the lid, the vial should be inverted and tapped to check for air bubbles. If there are any air bubbles present the sample must be retaken. Two vials should be filled per sample location.

Samples for analyses of organic chemicals should not be filtered. Samples for analyses of total organic halogens (TOX) and total organic carbon (TOC) are to be handled the same as samples to be analyzed for volatile organic compounds.

When collecting the samples for analyses of metals, the liquid will be filtered through a 0.45 micrometer filter prior to placement into the sample container. Samples for analyses of metals should be acidified to prevent precipitation of metals.

TABLE 4  
SAMPLING AND PRESERVATION REQUIREMENTS

EPA Method	Chemical Characteristic	Method Reference	Method Description	Con-tainer Type	Vol-ume (ml)	Pres-erv-ation	Max. Holding Time
EPA 602	Aromatic volatile organics compounds	EPA 600 1982	GC/PID	Glass	40	Cool 4°	14 days
EPA 601	Halogenated volatile organic compounds	EPA 601 1982	GC/HALL	Glass	40	Cool 4°	14 days
EPA 7196	Hexavalent chromium	SW 846 1986	Spectro-phometer	Plastic	1000	Cool 4°	24 hrs
EPA 6010	Total chromium	SW 846 1986	ICAP/AA	Plastic	1000	HNO <sub>3</sub>	6 mo
EPA 6010	Total cadmium	SW 846 1986	ICAP/AA	Plastic	1000	HNO <sub>3</sub>	6 mo
EPA 6010	Total zinc	SW 846 1986	ICAP/AA	Plastic	1000	HNO <sub>3</sub>	6 mo
EPA 300.0	Chloride	SW846 1986	Water Ex-trac-tion/IC	Plastic	50	Cool 4°	28 days
EPA 300.0	Nitrate	SW 846 1986	Water Ex-trac-tion/IC	Plastic	1000	H <sub>2</sub> SO <sub>4</sub>	14 days
EPA 9020	pH	SW 846 1986	pH Meter	Plastic	25	None	Field
EPA 9020	Total organic halogens	SW 846 1986	Microculo-metric Titration Detector	Glass	4x15	Cool 4° Sodium Sulfite	7 days
EPA 9060	Total organic carbon	SW 846 1986	Infrared Detector	Glass	4x15	Cool 4° HCL	28 days
EPA 9050	Specific conductance	SW 846 1986	Conductivity Meter	Plastic	25	None	Field

### 6.3.2 SAMPLE COLLECTION SEQUENCE

Sample collection will be performed in order of the volatilization sensitivity of the compounds of concern. The sequence of sampling for the Southern California Chemical Company facility is as follows:

1. EPA 601
2. EPA 602
3. Total organic halogens
4. Total organic carbons
5. Metals (Cd, Cu, Zn, Cr)
6. Hexavalent chromium
7. Chloride
8. Nitrate
9. pH
10. Specific conductance

Temperature, pH, and specific conductance will be measured in the field as outlined in section 6.3.

### 6.4 SAMPLE TRANSPORTATION

Department of Transportation (DOT) regulations will be strictly adhered to when commercial carriers are used to transport samples. The samples on this project will be transported to the laboratory under the custody of field collection personnel or a technician from the laboratory. All samples will be properly packed and maintained at proper temperatures (i.e., iced cooler) during transport. The method of shipment and any unusual circumstances pertaining to the shipment will be recorded on the chain-of-custody form.

### 6.5 SAMPLING FREQUENCY

40 CFR 265.92 requires that the EPA indicator chemicals (pH, TOC, TOX, cond) must be collected and analyzed in quadruplicate quarterly for at least one year and then semi-annually thereafter. Over two years of quarterly samples have been collected on the SCC project. Therefore, subsequently pH, TOC, TOX and conductivity will be collected and analyzed semi-annually.

Due to the elevated concentrations of organic compounds along the northern property boundary, monitoring wells MW-3, MW-4, MW-8, MW-9, MW-10, and MW-11 will be sampled quarterly. All other wells will be sampled semi-annually.

The site specific indicator chemicals (Cr, Cu, Cd, Zn, N, Cl) will be sampled quarterly for all wells. The well sampling frequency for each compound is summarized in Table 5.

**TABLE 5**  
**SAMPLING FREQUENCY**

Chemical Characteristic	Monitoring Well											
	MW- 1	MW- 2	MW- 3	MW- 4	MW- 4A	MW- 5	MW- 6	MW- 7	MW- 8	MW- 9	MW- 10	MW- 11
Organics Compounds (EPA 601 & 602)	SA	SA	Q	Q	SA	SA	SA	SA	Q	Q	Q	Q
Metal (Cr <sup>+6</sup> , Cr, Cd, Zn)	Q	Q	Q	Q	Q	Q	Q	Q	Q	Q	Q	Q
Chloride	Q	Q	Q	Q	Q	Q	Q	Q	Q	Q	Q	Q
Nitrate	Q	Q	Q	Q	Q	Q	Q	Q	Q	Q	Q	Q
EPA Indicator Groups (TOX, TOC, pH, Cond.)	SA	SA	SA	SA	SA	SA	SA	SA	SA	SA	SA	SA

Q = Samples for this chemical will be collected quarterly

SA = Samples for this chemical, group, or characteristic will be collected semi-annually

## 7 CHAIN OF CUSTODY

### 7.1 FIELD PROCEDURES

A critical aspect of sound sampling and analyses protocols is the maintenance of strict chain-of-custody procedures. Field chain-of-custody procedures include inventorying and record keeping during sample collection and shipment, through receipt by the laboratory.

#### 7.1.1 FIELD LOG BOOK

The following specific information will be entered in appropriate field log books:

- o Sample number assigned
- o Sampling location description--to include source(s)
- o Detailed description of sampling method, including a list of the settings on any equipment used in the sampling process
- o Date and time of sampling
- o Name(s) of individual taking the sample
- o Amount of sample collected or number of data values recorded
- o QA/QC procedures followed at the site, such as taking sample blanks, onsite field calibrations performed, etc.
- o If a composite, the frequency of sampling and the volume(s) of aliquot(s)
- o Flow information
- o Observations of any unusual events
- o Weather conditions
- o Field measurement such as temperature and pH

#### 7.1.2 CHAIN OF CUSTODY

Each sample then will be uniquely and completely labeled immediately following collection and a sampling chain-of-custody form (shown in Figure 3) will be prepared. Such forms will accompany the samples throughout the shipping and analytical process.

The chain-of-custody form will include:

- o Sample number
- o Date and time sample taken
- o Date and time sample submitted to the laboratory

## CHAIN OF CUSTODY RECORD

**SAMPLERS: (Signature)**

Phone: \_\_\_\_\_

SHIP TO:

**ATTENTION:** \_\_\_\_\_

Phone No. \_\_\_\_\_

Relinquished by: (Signature) Rec

Relinquished by: (Signature) Rec

Relinquished by: (Signature)	Rec
------------------------------	-----

Relinquished by: (Signature)	Rec
------------------------------	-----

## SHIPPING INFORMATION

Shipper \_\_\_\_\_

**Address** \_\_\_\_\_

Date Shipped \_\_\_\_\_

**Shipment Service** \_\_\_\_\_

Airbill No. \_\_\_\_\_

Cooler No. \_\_\_\_\_

Received by: (Signature)	Date/Time
--------------------------	-----------

Date/Time

Received by: (Signature)	Date/Time
--------------------------	-----------

Date/Time

Received by: (Signature)	Date/Time
--------------------------	-----------

Date/Time

Receive for laboratory by*: (Signature)	Date/Time
---	-----------

Date/Time

\*Analysis laboratory should complete, "sample condition upon receipt", section below, sign and return original (white) copy to KLEINFELDER, 17100 Pioneer Blvd., Suite 350, Artesia, CA 90701

[illegible]

**LAB INSTRUCTIONS:** Laboratory reports should reference and be billed by site ID# and contain the following:

- (1) summary of analytical methodology and QA work (blanks, spikes, duplicates)
- (2) dates for (a) sampling, (b) lab receipt, (c) extraction, (d) injection/analysis
- (3) detection limits for all constituents analyzed for and reporting of all constituents detected which were not specifically designated

(4) \_\_\_\_\_

(5) \_\_\_\_\_

**FIGURE**

3

**White - Kleinfelder**

Canary - Laboratory Courtesy Copy

### Pink - Sampler



- o Sample taken by (signature)
- o Sample matrix
- o Sample received by (signature)
- o Remarks (expected interferences, hazards, unusual events at the time of sampling)
- o Preservatives added (if any).

If more information on any sample is necessary, the appropriate field notebook pages will be referenced on the chain-of-custody forms.

Samples and data records will be maintained under proper holding conditions by the field chemist or technician until custody is relinquished to the recipient and formal documentation of such transfer is completed. A signed copy of the chain-of-custody form will be delivered to the appropriate task manager and kept on file for a minimum of three years.

### 7.1.3 CUSTODY SEALS

Custody seals will be affixed to the container at the time of sampling. Seals will be attached over the container lid in such a way that it will be necessary to break the seal when the sample is opened.

The sample custody seal will include:

- o Sample number
- o Name of collector
- o Date and time of samples

### 7.2 LABORATORY PROCEDURES

Laboratory chain-of-custody procedures are described in Appendix A and Appendix B, for the respective laboratories.

## 8 CALIBRATION PROCEDURES AND FREQUENCY

### 8.1 FIELD CALIBRATION PROCEDURES

Field measurements of temperature, pH and specific conductance will be periodically recorded for the purge water to document steady state sampling conditions. Field measurement of these parameters will be performed using the following protocols.

- o Prior to each round of sampling, all instruments will be calibrated per manufacturers' instructions using calibration solutions maintained at approximate ambient ground water temperature.
- o A calibration check will be performed at the beginning, middle, and end of each day, using calibration solutions at ambient field temperature.
- o Calibration solutions will span the range of field values obtained.
- o Probes will be rinsed thoroughly with deionized water between readings.

Calibration measurements will be recorded in the field notebook and submitted to the appropriate task manager for his review.

### 8.2 LABORATORY CALIBRATION PROCEDURES

Laboratory calibration procedures are provided in Appendix A and Appendix B, for the respective laboratories.

## 9 ANALYTICAL PROCEDURES

Chemical Research Laboratory will provide the primary analytical services for analyses of the groundwater samples collected for this project. A list of Chemical Research's procedures and protocols is given in Appendix A. Brown and Caldwell will provide analytical services for the analyses of duplicate or split samples performed for this project. A list of Brown and Caldwell's procedures and protocols is given in Appendix B.

## 10 DATA VALIDATION PROCEDURES

Data validation procedures are incorporated into various stages of the data generation process. Prior to issuing the draft laboratory report, all data are reviewed by laboratory QA/QC personnel (see Appendices A and B) for compliance with established procedures, protocols, and quality assurance objectives. Upon receiving the draft laboratory report, the appropriate Kleinfelder task manager prepares draft summary tables of the data, including summaries of the spike, duplicate, and blank samples, which are submitted as if they were normal well water samples. Quality control sample results are evaluated using established quality assurance criteria, and challenged when results are reported outside the acceptable ranges. Monitoring well results are reviewed in conjunction with knowledge of past monitoring data and hydrogeologic conditions, and challenged when reported results do not coincide with established trends. Bases for challenging the data will include, among others, the following:

1. Significant deviation from water-quality trends over time at the well, or
2. Significant deviation from nearby wells, especially during the first several rounds of analysis
3. For a given well, significant deviation from established ratios with concentrations of other sampled constituents with similar properties
4. Known cross-contamination, based upon results of QA/QC procedures
5. Known laboratory analytical or transcriptive error.

"Challenged" data are carefully reviewed by the laboratory, and are verified or discounted. A final laboratory report is then issued, and the results are summarized in tabular and graphical form.

## 11 INTERNAL QUALITY CONTROL CHECKS AND FREQUENCY

### 11.1 FIELD QUALITY CONTROL CHECKS

#### 11.1.1 TRAVEL BLANKS

Travel blanks are samples which will be prepared by the laboratory by filling representative glassware to be used for sample collection with known "organic free" water. These samples will be transported with the sample collection glassware and analyzed for evidence of systematic contamination from sample transport, glassware cleaning, and laboratory storage. One organic travel blank will be analyzed per day per transport container.

#### 11.1.2 FIELD BLANKS

To verify that the sampling system is not cross-contaminating well samples, field blanks will be collected by running distilled water through the system immediately following the normal cleaning procedures. This will be accomplished as follows:

- o Place the submersible pump in a stainless steel bucket which has been previously cleaned with steam and filled with distilled or deionized water.
- o Run 5 gallons of distilled water through the pump and line assembly (filling the bucket when necessary with water).
- o Collect water samples in appropriate sample containers from the discharge line.
- o Submit samples to laboratory for analysis.

The purpose of this sample will be to determine if the sampling equipment has any residual contamination that could influence the validity of the next well sample. Samples will be collected prior to the first well sample and then after every third well sampled as part of the SCC sampling.

#### 11.1.3 FIELD DUPLICATE SAMPLES

Duplicate samples are taken at each sampling site. In the case of 40 ml VOA vials, two vials are obtained per sample. This provides that if breakage or trouble with the testing equipment occurs, there is a backup sample for testing. This also allows a recheck on results if there is an inconsistency or if confirmation of results becomes necessary.

#### 11.1.4 SPIKE SAMPLES

Spike samples are samples of known concentration which are submitted "blind" (as if they were well samples) to evaluate the accuracy of laboratory results. A third laboratory will provide samples "spiked" with the compounds of concern. The calculated concentration for the spike will be verified by chemical analyses prior to submittal of the samples to the primary and secondary laboratories. A set of the spiked samples will be sent "blind" to both laboratories in another effort to verify the consistency of the data. The spiked samples for SCC will contain organic compounds in the 0.1 and 0.5 mg/l range and hexavalent chromium in the 1.0 mg/l range.

#### 11.1.5 SPLIT SAMPLES

Split samples are duplicate sets of samples "split" to both the primary and secondary laboratories. The duplicates are taken in sequence with one set going to each laboratory. On 30 percent of the monitoring wells, samples for organic analyses will be collected and "split" as part of this project. This will provide a check on the precision and accuracy of the laboratories. Comparison of the splits will provide a means of determining the confidence level of the data.

#### 11.2 LABORATORY QUALITY CONTROL CHECKS

Laboratory quality control checks are described in Appendix A and Appendix B, for the respective laboratories.

## 12 PERFORMANCE AND SYSTEMS AUDITS

The Quality Assurance Officer will monitor and audit the performance of the QA procedures outlined in the QAPP. The Quality Assurance Manager, or a qualified designee, will conduct field and office audits which will check that the information being gathered is reliable and of good quality. Audit reports will be submitted to the project manager following the completion of each audit evaluation.

### 12.1 FIELD/OFFICE AUDITS

The Quality Assurance Officer may schedule audits of field activities at various times to evaluate the execution of sample identification, sample control, chain-of-custody procedures, field documentation, instrument calibration, and field measurement and sampling operation.

Field documents pertaining to sample identification and control will be examined for completeness and accuracy. Field notebooks and field data forms will be reviewed to see that all entries are dated and signed and that the contents are legible and contain accurate and complete documentation of project activities. Because the notebook and field data forms provide the basis for reports written later, they will contain only facts and observations.

Sampling operations will be evaluated to determine if they are performed as stated in the project plan or as directed by the Project Manager. The QA officer will check to determine that the appropriate number of samples are being collected, that the samples are being placed in proper containers, and that proper preservation, packaging, and shipment protocol are being followed. The QA officer will also check to see that chain-of-custody procedures are being followed and that samples are being kept in custody at all times and are locked to prevent tampering.

Field measurement activities will be evaluated to determine if they are performed according to guidelines of this document. The QA officer will "spot check" various instruments for proper calibration and proper frequency of calibration, and will also check that the techniques utilized with these instruments are providing accurate data.

Once a field project has been completed, the individual files will be assembled, organized, and securely stored. The documents will be examined to determine whether all necessary items such as signatures, dates, and project codes are included. The QA officer will examine all documents and determine whether they are being handled and stored in the proper manner.

In addition to the formal audits performed, the project manager reviews product quality as draft documents are produced and checks that the project is being performed in accordance with approved quality assurance procedures. Prior to the production of a draft report, all work products will receive review by senior project staff. This review will include calculation briefs, test analyses, field measurements, graphs, tables, and any document which involves information generated from the field data.

## 12.2 LABORATORY AUDITS

Internal laboratory audits are described in Appendix A and Appendix B, for the respective laboratories.



## 13 PREVENTATIVE MAINTENANCE

### 13.1 FIELD PREVENTATIVE MAINTENANCE

Preventative maintenance for quality assurance includes those tasks that must be carried out to minimize downtime of the measurement systems.

Field procedures for preventative maintenance include the following:

- o Instruments for field measurements will be calibrated and checked before use.
- o Spare parts for instruments, such as probes, will be on hand in case of equipment failure.
- o When practical, backup equipment will be available.
- o Sufficient well construction materials will be on hand to account for monitoring well variability as dictated by geologic conditions.
- o Sampling locations and procedures will be identified and reviewed prior to field work.
- o Additional materials for potential additional sample preparation such as containers, caps, and forms will be available on-site.

Permanent records of the calibration and maintenance of each instrument will be maintained under the direction of the quality control officer.

### 13.2 LABORATORY PREVENTATIVE MAINTENANCE

Laboratory equipment downtime will be minimized by proper calibration and maintenance as discussed in Appendix A and Appendix B, for the respective laboratories.

## 14 DATA ASSESSMENT PROCEDURES

As part of the Quality Assurance Project Plan, routine procedures will be used to assess the precision, accuracy, and completeness of data for every type of measurement. In addition, an extensive review of field and analytical data will be conducted to check that quality control criteria have been met. Data assessment procedures to evaluate accuracy, precision, and completeness of laboratory data are described in the following sections.

### 14.1 ACCURACY

Accuracy is defined as the percent recovery for a spiked sample. A sample spike is prepared by adding a known amount of a reagent grade compound to the environmental sample. The compound added is the same as that being analyzed in the environmental sample. These spikes simulate the background and interferences found in the actual samples; the calculated percent recovery of the spike is taken as a measure of the accuracy of the total analytical method. When there is no change in volume due to the spike, it is calculated as follows:

$$P = \frac{(D-X) 100}{(T-X)} = \text{Percent Recovery}$$

X = Measured value of chemical concentration in the sample before spike is added.

D = Measured value of chemical concentration in the sample after the spike is added.

T = Assumed true value of chemical concentration in the sample after the spike is added.

Percent recovery criteria for each parameter group of interest are given in Appendix A and Appendix B, for the respective laboratories.

### 14.2 PRECISION

Precision is defined as the relative percent difference of matrix spike recoveries for multiple matrix spikes of the same sample (replicates), and is evaluated in terms of the relative percent difference. Because of the limited number of replicate samples that can be analyzed in environmental samples using gas chromatograph techniques, precision cannot be evaluated in terms of standard deviations. Consequently, "outlier" testing is not possible. However, the precision of an analytical method can be evaluated from internal laboratory and field duplicates by calculating the percent difference between the duplicate sample results:

$$PD = 2 \times \frac{(D1-D2)}{(D1+D2)} \times 100$$

PD = Percent Difference

D1 = First Sample Value

D2 = Second Sample Value (duplicated)

Precision criteria for each parameter of interest are presented in Appendix A.

### 14.3 COMPLETENESS

Completeness is described as the ratio of acceptable laboratory results to the total number of analyses performed. A completeness value of less than 95% indicates that corrective action is necessary in order to limit the number of defective results.

Criteria for defective results may include exceeded holding times, percent recoveries outside the limits in Appendix B, or unsatisfactory supporting data such as dates, locations, or sample identity numbers. An analysis of sample completeness will be conducted after each sampling round results are returned. Completeness is defined as:

$$C = \left( 1 - \frac{\text{number of defective results}}{\text{total number of requested analysis}} \right) 100$$

A C value less than 95% will indicate corrective action in order to avoid repeating similar problems in future sampling rounds.

### 14.4 ASSESSMENT

Field data will be assessed by evaluating adherence to the quality assurance program guidelines. Data collected historically in the study areas will be reviewed and used during the investigation. The kind of data to be used may include water level measurements, chemical data on water and soil, geologic subsurface descriptions, (e.g., drillers' logs) and interpretations of the hydrologic conditions of the study areas.

Each data point used in the investigation will be evaluated against the QAPP standards for a particular type of data collection. For example, a water-level measurement collected at a particular well will be assigned a high level of confidence if the data point is accompanied by information on the type of water level measuring device, the measuring point identification, pumping status of the measured well, construction details of the well, and the general pumping status of adjacent wells. If any of these data should be missing, the recorded, historical water level will be assigned a lower level of confidence, and may be rejected for the analysis of historical conditions.

Historical chemical data on the nature of soil or water conditions in the study areas will be similarly evaluated against the quality control procedures outlined in this document. Unless information is available on the method of sample collection, the analytical methods employed, and the quality assurance/quality control procedures used, the data point will be assigned a low level of confidence.

Data collected during the investigation should be collected according to the procedures outlined in this document. Any apparent data collection errors will be identified by evaluation of adherence to the QAP procedures and evaluation of the data compared to historical trends. If a data point appears to deviate from an anticipated trend, further investigations into the collection methodology and QA/QC procedures will be undertaken to resolve questionable data points. Pending the conclusion of these evaluations, the data point may either be accepted with a high or low level of confidence or rejected.

## 15 CORRECTIVE ACTION

The need for corrective action comes from several sources: equipment malfunction; failure of internal QA/QC checks; failure of performance or system audits; and noncompliance with QA requirements.

If measurement equipment or analytical methods fail QA/QC checks, the problem will immediately be brought to the attention of the Project Director, Project Manager, and QA Officer. If failure is due to equipment malfunction, the equipment will be repaired, precision and accuracy will be reassessed, and the analyses will be rerun, if practical.

A brief report of the corrective action will include statements on the following: 1) the malfunction-problem identified, 2) the steps and measures taken to remedy the malfunction, and 3) the recalibration of the instrumentation for precision and accuracy checks. This report will be submitted to the Project Manager and QA/QC Officer upon completion of the corrective action.

## 16 QUALITY ASSURANCE REPORTS TO MANAGEMENT

The final report and progress reports will include a section or statement that discusses and evaluates data quality and validity. At a minimum, the following information will be covered in the progress reports and the final report:

- o Assessment of measurement data precision, accuracy, and completeness.
- o Documentation of QA/QC practices.
- o Performance audit results.
- o System audit results.
- o Significant QA problems and recommended solutions.

Preparation of the quality assurance reports to management is the responsibility of the quality assurance officer. The quality assurance officer may assign portions of the report preparation to the appropriate task manager.

**APPENDIX A**

**LABORATORY QUALITY ASSURANCE PLAN**  
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**QUALITY ASSURANCE PROJECT PLAN**

PREPARED FOR: Kleinfelder

PROJECT: (50-1014-03)

CRL PROJECT MANAGER: \_\_\_\_\_

C. Aviles  
C. Aviles

CRL QA OFFICER: \_\_\_\_\_

S. Palubinskas (by SA)  
S. Palubinskas



## **Chemical Research Laboratories, Inc.**

### **CONTENTS**

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	PAGE	REVISION	DATE
1.0 PROJECT DESCRIPTION	1	0	5/03/88
2.0 PROJECT ORGANIZATION AND RESPONSIBILITY	1	0	5/03/88
3.0 QA OBJECTIVES	4	0	5/03/88
4.0 SAMPLING PROCEDURES	5	0	5/03/88
5.0 SAMPLE CUSTODY	5	0	5/03/88
6.0 ANALYTICAL METHODS, CALIBRATION PROCEDURES AND FREQUENCY	6	0	5/03/88
7.0 DATA REDUCTION, VALIDATION AND REPORTING	11	0	5/03/88
8.0 INTERNAL QUALITY CONTROL CHECKS	13	0	5/03/88
9.0 PERFORMANCE AND SYSTEM AUDITS	16	0	5/03/88
10.0 PREVENTATIVE MAINTENANCE	16	0	5/03/88
11.0 PROCEDURES FOR ASSESSING DATA PRECISION, ACCURACY AND COMPLETENESS	17	0	5/03/88
12.0 CORRECTIVE ACTION	18	0	5/03/88
13.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT	18	0	5/03/88

#### **APPENDIX A-1**

Definition and Procedure for the Determination of  
the Method Detection Limit





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Sec. No. 1.0

Revision No. 0

Date 5/03/88

Page 1 of 18

### **1.0 PROJECT DESCRIPTION**

The following Quality Assurance Project Plan (QAPP) describes the groundwater monitoring program designated for this project. Kleinfelder is the consultant firm performing the sampling for this project and managing the monitoring program.

The Consultant's strict confidentiality policy precludes disclosure of the monitoring program objectives. Therefore, the reasons for performing the monitoring, program objectives, and length of the program shall be addressed by the Consultant.

Quarterly testing shall be performed by Chemical Research Laboratories (CRL) upon request by Kleinfelder for the following parameters:

- Chloride
- Nitrate (expressed as nitrate and as nitrogen)
- Metals: Cadmium, Copper, Zinc, Total and Hexavalent Chromium
- Purgeable Halocarbons
- Purgeable Aromatics
- pH (Quadruplicate analysis)
- Total Organic Carbon (Quadruplicate analysis)
- Total Organic Halides (Quadruplicate analysis)
- Specific Conductance (Quadruplicate analysis)

### **2.0 PROJECT ORGANIZATION AND RESPONSIBILITY**

Project organization for analytical services performed by CRL staff is illustrated in Figure 1. Corresponding responsibilities are prescribed to ensure that sample handling and testing adheres to EPA protocol and serial data review. A description of these responsibilities follows:

**PROJECT MANAGER:** Monitors all phases of sample handling and processing from receipt of samples through report generation. Verifies project compliance prior to data release.

**SAMPLE CUSTODIAN:** Receives samples and accompanying documents. Records condition of samples and verifies information on the chain of custody with samples. Assigns and logs each sample batch with a unique accession number. Monitors and documents sample storage conditions. Dispenses samples to analysts.

**QA OFFICER:** Reviews quality assurance/quality control (QA/QC) procedures and initiates corrective actions. Prepares audit samples for assessing method, instrument, and analyst performance.



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Sec. No. 2.0  
Revision No. 0  
Date 5/03/88  
Page 2 of 18

**2.0 PROJECT ORGANIZATION AND RESPONSIBILITY - continued**

**ORGANIC SUPERVISOR:** Interprets, evaluates, and approves organic (gas chromatography) data for acceptability and accuracy. Supervises organic analysis staff. Performs instrument troubleshooting.

**GC CHEMIST:** Performs gas chromatographic (GC) analysis of both purgeable aromatics and halogenated compounds. Performs instrument preventative maintenance and instrument troubleshooting. Reviews data.

**INORGANIC SUPERVISOR:** Supervises inorganic staff performing the following analyses: Ion chromatography (IC), inductively coupled argon plasma (ICAP), general inorganic testing. Evaluates and approves inorganic data. Performs instrument troubleshooting.

**IC CHEMIST:** Performs chloride and nitrate (anion) analysis. Performs instrument preventative maintenance and troubleshooting. Reviews data.

**ICAP CHEMIST:** Performs metals analysis. Performs instrument preventative maintenance. Reviews data.

**INORGANIC ANALYSTS:** Perform pH, specific conductance, and sample preparation. Perform instrument preventative maintenance. Review data.

**TOC/TOX ANALYSTS:** Perform Total Organic Carbon (TOC) and Total Organic Halides (TOX) analyses. Perform instrument preventative maintenance and troubleshooting. Review data.



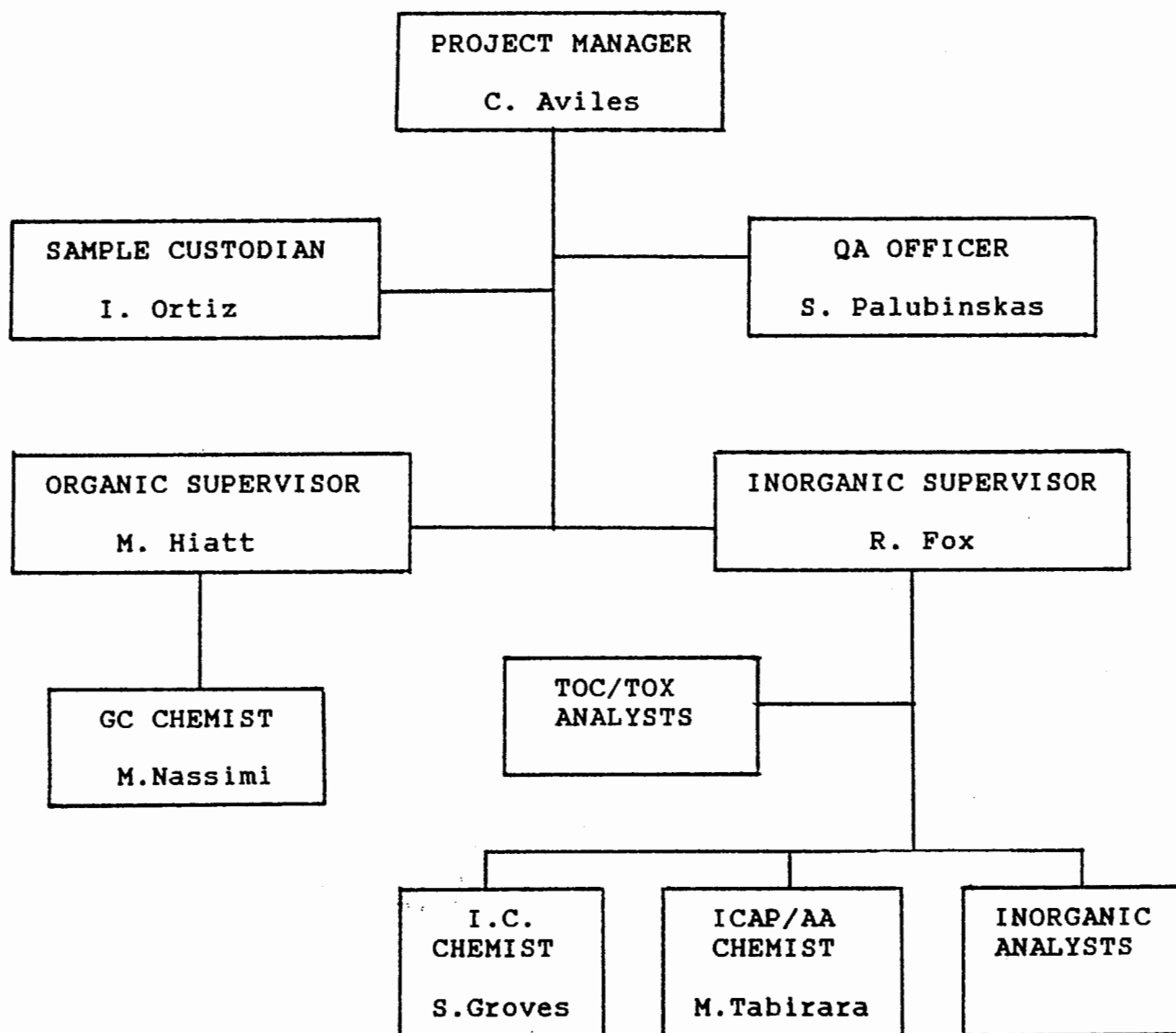
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Revision No. 0  
Date 5/03/88  
Page 3 of 18

**2.0 PROJECT ORGANIZATION AND RESPONSIBILITY - continued**



**Figure 1. Organization Chart**

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QA objectives for accuracy and precision of each parameter measured in this project are based upon demonstrated performance of the respective instrumentation and measurement system. These objectives focus on matrix spike/duplicate criteria consistent with the State of California Department of Health Services quality assurance requirements. These requirements include sample spiking and duplicate analysis for 10% of the samples analyzed. Table I lists QA acceptance limits for water samples.

**TABLE I****QA Acceptance Limits**

EPA METHOD	SPIKE PARAMETER	SPIKE RECOVERY LIMITS %	RELATIVE PERCENT DIFFERENCE (RPD) LIMITS
601 <sup>1</sup>	1,1-Dichloroethene	60 - 120	40
601 <sup>1</sup>	Trichloroethene	60 - 120	40
601 <sup>1</sup>	Chlorobenzene	60 - 120	40
602 <sup>1</sup>	Toluene	60 - 120	40
602 <sup>1</sup>	Xylene	60 - 120	40
602 <sup>1</sup>	Ethylbenzene	60 - 120	40
300.0 <sup>2</sup>	Chloride	87 - 119	12
300.0 <sup>2</sup>	Nitrate-Nitrogen	87 - 123	12
9060 <sup>3</sup>	Total Organic Carbon	79 - 119	20
9020 <sup>3</sup>	Total Organic Halides	56 - 127	25
6010 <sup>3</sup>	Cadmium	63.2 - 136	28
6010 <sup>3</sup>	Copper	57.7 - 127	37
6010 <sup>3</sup>	Total Chromium	47.3 - 159	47
6010 <sup>3</sup>	Zinc	52.8 - 144	36
7196 <sup>3</sup>	Hexavalent Chromium	60 - 120	40
9040 <sup>3</sup>	pH	N/A	+/- 0.3(units)*
9050 <sup>3</sup>	Specific Conductance	N/A	20%(umho/cm)*

\* RPD limits for duplicate analysis.

NOTE: Results are reported in mg/l except where noted.

<sup>1</sup>Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, 1982.<sup>2</sup>Methods for Chemical Analysis of Water and Wastes, Technical Add., 1984.<sup>3</sup>Test Methods for Evaluating Solid Waste (SW 846), Third Ed., 1986.



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Sec. No. 4.0  
Revision No. 0  
Date 5/03/88  
Page 5 of 18

#### 4.0 SAMPLING PROCEDURES

Sampling procedures shall be executed by Kleinfelder personnel and shall be described by Kleinfelder.

#### 5.0 SAMPLE CUSTODY

Each sample received by CRL shall be assigned a unique laboratory number consisting of the year, Julian date, and time. This accession number along with pertinent sampling information shall be logged into CRL's computer database system. Other information recorded includes the date and time of sampling, sample description, location and required tests. Following log-in, samples are stored in a walk-in refrigeration unit maintained at 4°C.

All sample log-in, storage and chain-of-custody documentation is the responsibility of the sample custodian, Mr. Ike Ortiz. The sample custodian is authorized to sign for incoming samples, retain documents of shipment, and verify data entered into the sample custody records. Additionally, the sample custodian shall ensure that sample storage is secure and maintained at the proper temperature. Samples removed from storage shall be signed over to the analyst by the sample custodian and shall be accounted for daily. Completed chain-of-custody documentation is included with the final report. A bound permanent logbook is maintained by the sample custodian for documenting the following:

- 1 - Date and time of sample receipt.
- 2 - Source of sample.
- 3 - Sample accession number.
- 4 - Parameters to be analyzed.
- 5 - Matrix.
- 6 - Date reported.
- 7 - Final disposition of sample  
(30 days after report issuance).



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Sec. No. 6.0  
Revision No. 0  
Date 5/03/88  
Page 6 of 18

## 6.0 ANALYTICAL METHODS, CALIBRATION PROCEDURES AND FREQUENCY

A standard calibration check is performed daily for each instrument along with reagent water blanks. The diluted standards are verified by EPA reference standards or standards otherwise traceable to the EPA or National Bureau of Standards. Continuing calibration verification is plotted on control charts for each contaminant analyzed. A summary of calibration procedures used in EPA methods assigned for this project is contained in Table II.

### 6.1 GAS CHROMATOGRAPHY

Daily analysis of standards, previously verified with EPA or NBS standards, are used to adjust the sensitivity and selectivity of the analytical system for each compound being analyzed. Calibration of the chromatographic system is accomplished by analyzing standards at a minimum of three concentration levels for each analyte. The low level standard is at or near the established detection limit. The medium and high level standards are at concentrations that correspond to the expected range of sample concentrations. A three point curve establishes the working range of the GC detector. Standards are validated daily with the use of QC check solutions.

The results of standard calibrations (low, medium, and high ranges) for each analyte are tabulated with respect to response versus concentration. The ratio between response and concentration, known as response factor (RF), can be used to prepare a calibration curve for each compound. Alternately, if the RF is constant (less than 10% relative standard deviation) over the working range it can be assumed that linearity exists and the average RF can be used in place of the daily calibration curve. Response factor calculation is shown in the following equation:

$$RF = \frac{(A_s) \times (C_{is})}{(A_{is}) \times (C_s)}$$

$A_s$  = Area of sample.

$C_{is}$  = Concentration of internal standard.

$A_{is}$  = Area of internal standard.

$C_s$  = Concentration of sample.

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Table II

## Summary of Calibration Procedures

EPA TEST (DESCRIPTION)	CALIBRATION PROCEDURE	CALIBRATION FREQUENCY	REFERENCE
EPA 300.0 (Chloride & Nitrate)	Establish linearity over a concentration range defined by a 3 point standard curve.	Calibrate each time response or retention time for any analyte varies more than $\pm 10\%$ of expected value.	Stock standard prepared from ACS reagent grade materials.
EPA 6010 (Total Metals)	Establish linearity over a concentration range defined by a 3 point standard curve.	Calibrate each time check standard results vary more than $\pm 10\%$ of expected value.	Stock standards prepared from high purity grade chemicals.
EPA 7196 (Hexavalent Chromium)	Establish linearity over a concentration range defined by a 3 point standard curve.	Calibrate daily.	Stock standards prepared from high purity grade chemicals.
EPA 601 (Purgeable Halocarbons)	Establish linearity over a concentration range defined by a 3 point standard curve.	Calibrate daily.	Analyzed stock standards are purchased.
EPA 602 (Purgeable Aromatics)	Establish linearity over a concentration range defined by a 3 point standard curve.	Calibrate daily.	Analyzed stock standards are purchased.
EPA 9040 (pH)	Optimize instrument slope to achieve $\pm$ .05 pH units between two standards whose range brackets the sample reading.	Calibrate daily.	Standard buffers are purchased.
EPA 9060 (TOC)	Establish linearity over a concentration range defined by a 3 point standard curve.	Calibrate daily.	Stock standard prepared from primary standard grade chemical.
EPA 9020 (TOX)	Establish linearity over a concentration range defined by a 3 point standard curve.	Calibrate daily.	Stock standards prepared from ACS reagent grade chemicals.
EPA 9050 (Specific Conductance)	Determine cell constant.	Calibrate daily.	Reference standard prepared from ACS reagent grade chemicals.



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Revision No. 0

Date 5/03/88

Page 8 of 18

Calibration standards, validated by reference standards, are prepared and analyzed daily and are monitored for stability. As with most analytical techniques, the solutions are prepared at three different concentrations to demonstrate and verify the operating range of the instrument. In addition to calibration standards, quality control (QC) is monitored by the analysis of duplicate samples, spike samples, QC check samples, and blanks every tenth sample. Calibration drift, within acceptable control limits is verified and recalibration is performed every 20 samples.

QC procedures are exercised throughout the entire analytical process. One of the control parameters monitored is sample interference. Samples exhibiting interference are reanalyzed using corrections determined by interference check sample analysis. Table III lists potential analyte concentration equivalents arising from interference. The ICAP interference check is performed at the beginning and end of each sample batch analysis run, or a minimum of twice per 8 hour working shift, whichever is more frequent. If an interference cannot be resolved by sample dilution, buffering, or matrix matching, standard addition procedures are used.

### **6.3 ION CHROMATOGRAPHY**

A working calibration curve is determined daily for each analyte based on injection of a minimum of three standard concentration levels. Response factors (peak height versus concentration) and retention times are recorded for each analyte from initial daily calibration. The calibration curve is verified after every twenty samples or when the anion eluent is changed. Standard concentration ranges are selected to bracket expected sample concentrations and are within demonstrable linearity studies for each analyte under specified instrument operating parameters. New calibration curves are prepared if response factors or retention times vary from expected values by more than +/- 10%.

### **6.4 TOTAL ORGANIC HALIDES**

Total Organic Halides are determined through a two step process of adsorption onto activated carbon followed by pyrolytic conversion to a titratable species and measured by a microcoulometric detector. The adsorption efficiency of each newly-prepared batch of carbon is tested by running duplicates of the adsorption-efficiency standard along with duplicates of the blank standard. Acceptable net recovery is defined as 5% of the standard value. Daily calibration requires analysis of two method blanks followed by a method blank between each group of eight pyrolysis determinations. Next, instrument calibration standards are run in duplicate and repeated after each group of eight pyrolysis determinations. Response of these calibration checks must be within 3% of the calibration standard value.



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Table III

**Analyte Concentration Equivalents Arising From Interference  
At The 100 mg/L Level**

Analyte	Wavelength (nm)	Interferent <sup>a,b</sup>									
		Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Tl	V
Aluminum	308.215	—	—	—	—	—	—	0.21	—	—	1.4
Antimony	206.833	0.47	—	2.9	—	0.08	—	—	—	0.25	0.45
Arsenic	193.696	1.3	—	0.44	—	—	—	—	—	—	1.1
Barium	455.403	—	—	—	—	—	—	—	—	—	—
Beryllium	313.042	—	—	—	—	—	—	—	—	0.04	0.05
Boron	249.773	0.04	—	—	—	0.32	—	—	—	—	—
Cadmium	226.502	—	—	—	—	0.03	—	—	0.02	—	—
Calcium	317.933	—	—	0.08	—	0.01	0.01	0.04	—	0.03	0.03
Chromium	267.716	—	—	—	—	0.003	—	0.04	—	—	0.04
Cobalt	228.616	—	—	0.03	—	0.005	—	—	0.03	0.15	—
Copper	324.754	—	—	—	—	0.003	—	—	—	0.05	0.02
Iron	259.940	—	—	—	—	—	—	0.12	—	—	—
Lead	220.353	0.17	—	—	—	—	—	—	—	—	—
Magnesium	279.079	—	0.02	0.11	—	0.13	—	0.25	—	0.07	0.12
Manganese	257.610	0.005	—	0.01	—	0.002	0.002	—	—	—	—
Molybdenum	202.030	0.05	—	—	—	0.03	—	—	—	—	—
Nickel	231.604	—	—	—	—	—	—	—	—	—	—
Selenium	196.026	0.23	—	—	—	0.09	—	—	—	—	—
Silicon	288.158	—	—	0.07	—	—	—	—	—	—	0.01
Sodium	588.995	—	—	—	—	—	—	—	—	0.08	—
Thallium	190.864	0.30	—	—	—	—	—	—	—	—	—
Vanadium	292.402	—	—	0.05	—	0.005	—	—	—	0.02	—
Zinc	213.856	—	—	—	0.14	—	—	—	0.29	—	—

<sup>a</sup> Dashes indicate that no interference was observed even when interferents were introduced at the following levels:

Al - 1000 mg/L,

Ca - 1000 mg/L,

Cr - 200 mg/L,

Cu - 200 mg/L

Fe - 1000 mg/L

Mg - 1000 mg/L,

Mn - 200 mg/L,

Ti - 200 mg/L,

V - 200 mg/L

<sup>b</sup> The figures recorded as analyte concentrations are not the actual observed concentrations; to obtain those figures, add the listed concentration to the interferent figure.

SOURCE: SW 846, 1986



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Sec. No. 6.5

Revision No. 0

Date 5/03/88

Page 10 of 18

**6.5 TOTAL ORGANIC CARBON**

The Total Organic Carbon Analyzer uses injection loops of varying volumes to analyze samples in three ranges of concentrations. For each range the instrument is calibrated with three calibration standards over the specific range. A water blank is analyzed with each calibration. Calibration check standards are run after every ten samples and at the end of each batch. The instrument is recalibrated at the beginning of a batch or when standard drift exceeds 10% of the calibration standard value.

**6.6 SPECIFIC CONDUCTANCE**

Calibration of a self-contained conductivity instrument consists of determining the cell constant at 25°C. The resistance of a 0.01 N KCl at 25°C is measured and the cell constant C is computed as:

$$C = (0.001413) (R_{KCl}) [1 + 0.0191 (t - 25)]$$

where:  $R_{KCl}$  = measured resistance, ohms

$t$  = observed temperature, °C

The cell constant is used in calculating all sample conductivities. A check standard is analyzed after 15 samples and at the beginning and end of a sample batch.

**6.7 pH**

The pH instrument/electrode system is calibrated over a range determined by two points that are three or more pH units apart and bracket the expected pH value of the samples. Repeated adjustments on are made until readings are within 0.05 pH units of the buffer solution values, respectively. Check standards are measured after every ten samples.

**6.8 COLORIMETRY**

Colorimetric determinations employ a spectrophotometer or filter photometer equipped to measure wavelengths within the light transmittance region specific to the compound being measured. Calibration involves a minimum three point curve of response versus concentration designed to bracket sample concentrations within the linear working range of the method. Calibration is verified after every twenty samples and should be within +/- 2% of check standard values.



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Sec. No. 7.0  
Revision No. 0  
Date 5/03/88  
Page 11 of 18

## 7.0 DATA REDUCTION, VALIDATION AND REPORTING

The data reduction scheme for ensuring valid data includes four levels of review. Each level commands specific action to prevent the unqualified release of erroneous data and to correct any problems discovered during the review process.

The first level of data review is with the analyst at the bench level. The analyst notes any anomalies observed during testing and verifies batch acceptability using accepted precision ranges. Data that does not meet acceptable precision criteria is immediately evaluated by the supervising chemist. Upon verification of correct calculations, acceptable instrument performance, and appropriate methodology, the standards are reanalyzed and/or reprepared and analyzed. Once the standard has performed acceptably, the instrument is recalibrated and the sample reanalyzed. If the data is then within accepted precision criteria, a representative portion of the sample batch will be reevaluated.

The second level of data review is with the supervising chemist who checks the data for completeness, consistency, and conformance with QA criteria. Any deviations in precision, calibration data, or laboratory blank monitoring will be evaluated on a case by case basis. Noncompliance with precision criteria will result in sample batch evaluation described in the first level of data review.

The third level of data review is exercised by the QA coordinator who performs additional statistical evaluations of data acceptability. Any discrepancies in spike recovery, precision criteria, or instrument performance result in a system check to identify the source of any deviations. The QA Coordinator collaborates with the supervising chemist in designating additional corrective action.

The fourth level of quality assurance is accomplished through the laboratory director. Weekly QA reports are reviewed at this level and are used in determining whether completed data is ready for release. Data that does not meet reporting requirements of less than three standard deviations from the mean established for any parameter may not be reported and is subject to reanalysis.

The principal criteria used to assess data integrity is the accuracy and precision of QC audits, quality assurance determinations (within each sample batch analysis), and thorough investigation of potential interferences. This multiple review process independently examines one or more criterion of data integrity and finally results in an assemblage of all analytical data which is evaluated prior to approval.



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Sec. No. 7.0  
Revision No. 0  
Date 5/03/88  
Page 12 of 18

## 7.1 DETECTION LIMITS

The guidelines described in Figure 3. summarize requirements for detection limit confidence levels--described in Appendix A-1. Detection limit studies are conducted on each instrument to determine confidence levels for the analytical method under evaluation. Figure 3 represents the reliability of measurements expressed in terms of sigma (standard deviation). Sigma denotes the precision of measurement for a given concentration.

The limit of detection is identified as sigma but is not the value established for data reporting. Detection limit reporting is actually based on the limit of quantitation (LOQ) and is defined as ten times the value of sigma. The LOQ defines the analyte concentration level at which relative confidence is approximately 30% with a probability of 95%.

---

Analyte conclusion  
in units of sigma

$(S_t - S_b)$	Reliability
< 3 sigma	Region of questionable detection (therefore, unacceptable)
3 sigma	Limit of Detection
3 - 10 sigma	Region of less certain quantitation
10 sigma	Limit of Quantitation
> 10 sigma	Region of quantitation

---

$S_t$  = Total value measure for the sample.  
 $S_b$  = Value for the blank.  
Sigma = One standard deviation.

---

Figure 3. Guidelines for reporting data based on detection limit.



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Sec. No. 7.2  
Revision No. 0  
Date 5/03/88  
Page 13 of 18

## 7.2 DATA REPORTING

Data reporting is an extended activity beginning with a department report of reviewed data, compiled into a complete data package, followed by QA department approval and finally by Laboratory Director review and approval. Upon completion of data review at all levels and subsequent clerical preparation and final typographical review, reports are signed by the Laboratory Director and issued to clients, respectively.

## 8.0 INTERNAL QUALITY CONTROL CHECKS

Internal quality control conducted at CRL focuses on ensuring that each chemical measurement has the highest probability of being acceptable in terms of precision and accuracy. Internal QC includes blank, spike, duplicate and reagent monitoring as described in the following sections.

### 8.0.1 METHOD BLANK

A method blank is used to monitor the system for interferences and contamination from glassware, reagents, etc. A blank is included with each batch of extractions and digestions prepared. The method blank is a distilled water blank that undergoes complete extraction, concentration, and analysis as the samples analyzed in that batch.

### 8.0.2 DUPLICATES AND SPIKES

Matrix spikes and matrix spike duplicates are used in each batch with a frequency of 10% or with each different sample matrix-- whichever is more frequent. Samples are spiked at concentrations twice the level found in the sample or at ten times the method detection limit. Recommended spiking levels for metals are listed in Table IV. Spiked samples that do not meet established precision criteria are evaluated further under CRL's data validation protocol.

As part of QC monitoring, a sample from each batch is split and both portions are analyzed to ensure data quality. This is the basis for precision monitoring within every batch of ten samples analyzed.

### 8.0.3 SPLIT SAMPLES

Samples submitted to CRL's Garden Grove facility are selected at random, split and analyzed by one or more of CRL's branch facilities. This activity not only identifies potential deficiencies within the participating technical sections, but also serves as an indicator of good performance and data reproducibility.

### 8.0.4 QUALITY CONTROL SAMPLES

CRL participates in the NPDES check sample program and also obtains EPA check sample solutions for internal quality monitoring and analysis technique validation.

**Chemical Research Laboratories, Inc.**

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Element	For ICP/AA (ug/L)		For Furnace AA (ug/L)		Other <sup>(2)</sup> (ug/L)
	Water	Soil	Water	Soil	
Aluminum	2,000	*			
Antimony	500	500	100	100	
Arsenic	2,000	2,000	40	40	
Barium	2,000	2,000			
Beryllium	50	50			
Cadmium	50	50	5	5	
Calcium	*	*			
Chromium	200	200			
Cobalt	500	500			
Copper	250	250			
Iron	1,000	*			
Lead	500	500	20	20	
Magnesium	*	*			
Manganese	500	500			
Mercury					1
Nickel	500	500			
Potassium	*	*			
Selenium	2,000	2,000	10	10	
Silver	50	50			
Sodium	*	*			
Thallium	2,000	2,000	50	50	
Vanadium	500	500			
Zinc	500	500			
Cyanide					100

NOTE: Elements without spike levels and not designated with an asterisk, must be spiked at appropriate levels.

<sup>1</sup>The levels shown indicate concentrations in the final digestate of the spiked sample (200 mL final volume).

<sup>2</sup>Spiking level reported is for both water and soil/sediment matrices.

\* No spike required.

SOURCE: sw 846, 1986



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Sec. No. 8.0.5  
Revision No. 0  
Date 5/03/88  
Page 15 of 18

### 8.0.5 CALIBRATION STANDARDS AND DEVICES

Whenever possible, standards are maintained traceable to U.S. EPA or NBS standards. EPA traceable standards are available directly from the EPA Quality Assurance Material Bank (MB-8) or Supelco. NBS standards are available from the National Bureau of Standards.

A standard logbook is used to document the preparation of standards and provide a means to trace each solution to the starting materials. Each entry is dated and signed by the preparer. The following information is documented for each standard:

- 1 - Lot number and manufacturer
- 2 - Concentration of each compound or solution
- 3 - Final volume of solution
- 4 - Concentration of solution components
- 5 - Name of solution
- 6 - Results of parallel testing

Standards are labeled by: 1) numbered standard log, 2) page number, and 3) entry number. Individual entries are described in reagent sections of CRL Standard Operating Procedures describing standard solutions required for analysis.

The final check of standard viability is analysis and subsequent comparison against a certified traceable standard independent of the prepared working standard.

### 8.0.6 STABILITY OF CONTROLS AND STANDARDS

Standards prepared for wet analysis are prepared and stored according to the method employed. Extreme care is taken to see that no standards are used after their expiration date.

### 8.0.7 VOLATILE ORGANIC STANDARDS

Stock solutions for volatile organics are not held for more than 30 days. Working calibration standards are prepared fresh weekly. A notation in the standard log is entered upon discarding the standard solution.

### 8.0.8 SEMI-VOLATILE ORGANICS STANDARDS

Stock solutions for semi-volatile organics are not held for more than 90 days. Dilutions below 1 ppm are not held more than 30 days. A notation in the standard log is entered upon discarding the standard solution.



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Sec. No. 8.0.9

Revision No. 0

Date 5/03/88

Page 16 of 18

### 8.0.9 GENERAL PRECAUTIONS

Stock and working solutions must be freshly prepared as often as required by their stability, and must be checked regularly for signs of deterioration, e.g. discoloration, formation of precipitates, and changes in concentration.

### 8.0.10 REAGENTS

Laboratory reagent water that meets the requirements of Type II water, as described in ASTM Part 31, is prepared daily. Blanks are routinely analyzed and accompany each batch tested.

High purity reagents are purchased as dictated by each test method and are documented by batch, lot number, and supplier as well as time period of laboratory use (date opened, date depleted).

## 9.0 PERFORMANCE AND SYSTEM AUDITS

CRL performance auditing includes blind and reference sample testing, quality control chart evaluation, standards monitoring and instrument calibration monitoring.

In-house sample audits are administered as a means of independent quality checks. These audit samples are prepared by a department supervisor or by the QA Coordinator and submitted to the analyst as part of the daily assignment schedule. Upon evaluation of the results, the analytical performance is discussed with the analyst and improvements are initiated as needed. Satisfactory performance in these audits provides verification that methodology is both effective and routinely adhered to.

Reference samples, that are independently requested from EPA or submitted by clients as blind samples, are used in CRL's internal auditing practices for groundwater monitoring programs contracted to CRL.

Standards and instrument monitoring are part of the daily routine operating procedures employed prior to performing analyses.

Quality control charts which measure fluctuations in data trends are evaluated monthly for precision/accuracy compliance as well as changes in instrument or method performance.

## 10.0 PREVENTATIVE MAINTENANCE

Preventative maintenance is a part of CRL operations protocol and is documented in maintenance logs assigned for each instrument.





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Sec. No. 10.0  
Revision No. 0  
Date 5/03/88  
Page 17 of 18

**10.0 PREVENTATIVE MAINTENANCE - continued**

Instrument servicing is scheduled according to prescribed manufacturer recommendations, or sample throughput--whichever is more frequent. In addition to existing service contracts for instrumentation, CRL has sufficient equipment to continue analyses in the event that instrument problems are encountered. In addition to backup instrumentation, a supply of spare parts such as gas chromatography columns, fittings, septums; atomic absorption lamps, mirrors, diaphragms; graphite furnace tubes; ICAP tubing; cold vapor glassware; hydride generation accessories and other ancillary equipment are maintained.

**11.0 PROCEDURES FOR ASSESSING DATA PRECISION, ACCURACY AND COMPLETENESS**

Control charts provide a useful tool in assessing QC efforts through graphical displays of a parameter and its variability over time. The parameter plotted is related to control sample testing, either directly in terms of concentration or indirectly in terms of derived information such as concentration mean (arithmetic) or range of concentration. Control charts take into consideration the following:

- 1- System is in control before beginning of control charting.
- 2- Number of control samples run.
- 3- Number of runs analyzed (greater than 10).
- 4- Parameters to be plotted against time.
- 5- Statistical basis for assigning warning and rejection limits.
- 6- Types of shifts, trends, or biases exhibited.

Unless otherwise noted, CRL control chart ranges span  $\pm 2$  and  $\pm 3$  standard deviations (S.D.). Two S.D. define CRL's warning limit for data acceptance. Three S.D. define CRL's control limit which if exceeded, requires reanalysis of all samples analyzed in the batch that exceeded 3 S.D. A gaussian distribution identifies 5% outliers referenced from 2 S.D. Quality control results (of known material) outside of the 2 S.D. control range must be reviewed by the supervisor responsible for the department generating the data in question: An evaluation of the datum in question, relevant QC information and interferences present determine whether these data are acceptable. Six successive points within 2 S.D. on the same side ( $\pm$ ) of the arithmetic mean require analyses to be stopped and the problem evaluated.

Accuracy and precision measures are routinely practiced and evaluated with each ten samples analyzed or each different sample matrix--whichever is more frequent.



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Sec. No. 12.0  
Revision No. 0  
Date 5/03/88  
Page 18 of 18

## 12.0 CORRECTIVE ACTION

Corrective action is dictated by the type and extent of out-of-control event encountered. Out-of-control events are defined as quality assurance data exceeding 3 S.D. or six successive points within 2 S.D. on the same side (+/-) of the arithmetic mean.

Corrective action may be initiated and carried out by non-supervisory staff, but final approval and data review by management is necessary to report any information. All potentially affected data must be thoroughly reviewed for acceptance or rejection.

Out-of-control events are documented as to the nature of the incident and the corrective actions taken to set the system back "in control." A corrective action report, to be signed by the section supervisor and quality assurance officer is required to document any actions taken on quality control results outside a two standard deviation range. The documentation must include:

- 1 - Where the out-of-control incident occurred (department and test name).
- 2 - Date of occurrence.
- 3 - Corrective action taken.
- 4 - Initials of operating chemist, supervisor and QA officer.

This information is filed with the control charts and an entry outside of two standard deviations is accompanied by a corresponding corrective action form.

## 13.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

Project quality assurance reports are prepared weekly by the Quality Assurance Officer and submitted to the Laboratory Director. These reports include data accuracy, precision, and completeness. All audits, QA problems, and corrective action measures performed during the week are included in these reports.

## Appendix A - 1

### Definition and Procedure for the Determination of the Method Detection Limit

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero and determined from analysis of a sample in a given matrix containing analyte.

#### Scope and Application

This procedure is designed for applicability to a wide variety of sample types ranging from reagent (blank) water containing analyte to wastewater containing analyte. The MDL for an analytical procedure may vary as a function of sample type. The procedure requires a complete, specific and well defined analytical method. It is essential that all sample processing steps of the analytical method be included in the determination of the method detection limit.

The MDL obtained by this procedure is used to judge the significance of a single measurement of a future sample.

The MDL procedure was designed for applicability to a broad variety of physical and chemical methods. To accomplish this, the procedure was made device- or instrument-independent.

#### Procedure

1. Make an estimate of the detection limit using one of the following:
  - (a) The concentration value that corresponds to an instrument signal/noise ratio in the range of 2.5 to 5. If the criteria for qualitative identification of the analyte is based upon pattern recognition techniques, the least abundant signal necessary to achieve identification must be considered in making the estimate.
  - (b) The concentration value that corresponds to three times the standard deviation of replicate instrumental measurements for the analyte in reagent water.
  - (c) The concentration value that corresponds to the region of the standard curve where there is a significant change in sensitivity at low analyte concentrations, i.e., a break in the slope of the standard curve.
  - (d) The concentration value that corresponds to known instrumental limitations.

It is recognized that the experience of the analyst is important to this process. However, the analyst must include the above considerations in the estimate of the detection limit.

2. Prepare reagent (blank) water that is as free of analyte as possible. Reagent or interference free water is defined as a water sample in which analyte and interferent concentrations are not detected at the method detection limit of each analyte of interest. Interferences are defined as systematic errors in the measured analytical signal of an established procedure caused by the presence of interfering species (interferent). The interferent concentration is presupposed to be normally distributed in representative samples of a given matrix.
3. (a) If the MDL is to be determined in reagent water (blank), prepare a laboratory standard (analyte in reagent water) at a concentration which is at least equal to or in the same concentration range as the estimated MDL. (Recommend between 1 and 5 times the estimated MDL.) Proceed to Step 4.

- (b) If the MDL is to be determined in another sample matrix, analyze the sample. If the measured level of the analyte is in the recommended range of one to five times the estimated MDL, proceed to Step 4.

If the measured concentration of analyte is less than the estimated MDL, add a known amount of analyte to bring the concentration of analyte to between one and five times the MDL. In the case where an interference is coanalyzed with the analyte:

If the measured level of analyte is greater than five times the estimated MDL, there are two options:

- (1) Obtain another sample of lower level of analyte in same matrix if possible.
  - (2) The sample may be used as is for determining the MDL if the analyte level does not exceed 10 times the MDL of the analyte in reagent water. The variance of the analytical method changes as the analyte concentration increases from the MDL, hence the MDL determined under these circumstances may not truly reflect method variance at lower analyte concentrations.
4. (a) Take a minimum of seven aliquots of the sample to be used to calculate the MDL and process each through the entire analytical method. Make all computations according to the defined method with final results in the method reporting units. If blank measurements are required to calculate the measured level of analyte, obtain separate blank measurements for each sample aliquot analyzed. The average blank measurement is subtracted from the respective sample measurements.
- (b) It may be economically and technically desirable to evaluate the estimated MDL before proceeding with 4a. This will: (1) prevent repeating this entire procedure when the costs of analyses are high and (2) insure that the procedure is being conducted at the correct concentration. It is quite possible that an incorrect MDL can be calculated from data obtained at many times the real MDL even though the background concentration of analyte is less than five times the calculated MDL. To insure that the estimate of the MDL is a good estimate, it is necessary to determine that a lower concentration of analyte will not result in a significantly lower MDL. Take two aliquots of the sample to be used to calculate the MDL and process each through the entire method, including blank measurements as described above in 4a. Evaluate these data:
- (1) If these measurements indicate the sample is in the desirable range for determining the MDL, take five additional aliquots and proceed. Use all seven measurements to calculate the MDL.
  - (2) If these measurements indicate the sample is not in the correct range, reestimate the MDL, obtain new sample as in 3 and repeat either 4a or 4b.

5. Calculate the variance ( $S^2$ ) and standard deviation ( $S$ ) of the replicate measurements, as follows:

$$S^2 = \frac{1}{n-1} \left[ \sum_{i=1}^n X_i^2 - \frac{\left( \sum_{i=1}^n X_i \right)^2}{n} \right]$$

$$S = (S^2)^{1/2}$$

where: the  $x_i$ ,  $i = 1$  to  $n$  are the analytical results in the final method reporting units obtained from the  $n$  sample aliquots and  $\sum_{i=1}^n X_i^2$  refers to the sum of the  $X$  values from  $i = 1$  to  $n$ .

6. (a) Compute the MDL as follows:

$$MDL = t_{(n-1, 1-\alpha = .99)} (S)$$

where:

MDL = the method detection

$t_{(n-1, 1-\alpha = .99)}$  = the students' t value appropriate for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom. See Table.

S = standard deviation of the replicate analyses.

- (b) The 95% confidence limits for the MDL derived in 6a are computed according to the following equations derived from percentiles of the chi square over degrees of freedom distribution ( $X^2/df$ ) and calculated as follows:

$$MDL_{LCL} = 0.69 MDL$$

$$MDL_{UCL} = 1.92 MDL$$

where  $MDL_{LCL}$  and  $MDL_{UCL}$  are the lower and upper 95% confidence limits respectively based on seven aliquots.

7. Optional iterative procedure to verify the reasonableness of the estimated MDL and calculated MDL of subsequent MDL determinations.
- (a) If this is the initial attempt to compute MDL based on the estimated MDL in Step 1, take the MDL as calculated in Step 6, spike in the matrix at the calculated MDL and proceed through the procedure starting with Step 4.
- (b) If the current MDL determination is an iteration of the MDL procedure for which the spiking level does not permit qualitative identification, report the MDL as that concentration between the current spike level and the previous spike level which allows qualitative identification.
- (c) If the current MDL determination is an iteration of the MDL procedure and the spiking level allows qualitative identification, use  $S^2$  from the current MDL calculation and  $S^2$  from the previous MDL calculation to compute the F ratio.

$$\text{if } \frac{S_A^2}{S_B^2} < 3.05$$

then compute the pooled standard deviation by the following equation:

$$S_{\text{pooled}} = \left[ \frac{6S_A^2 + 6S_B^2}{12} \right]^{1/2}$$

if  $\frac{S_A^2}{S_B^2} > 3.05$ , respoke at the last calculated MDL and process the samples through the procedure starting with Step 4.

- (c) Use the  $S_{\text{pooled}}$  as calculated in 7b to compute the final MDL according to the following equation:

$$MDL = 2.681 (S_{\text{pooled}})$$

where 2.681 is equal to  $t_{(12, 1-\alpha = .99)}$ .

- (d) The 95% confidence limits for MDL derived in 7c are computed according to the following equations derived from percentiles of the chi squared over degrees of freedom distribution.

$$MDL_{LCL} = 0.72 MDL$$

$$MDL_{UCL} = 1.65 MDL$$

where LCL and UCL are the lower and upper 95% confidence limits respectively based on 14 aliquots.

## Reporting

The analytical method used must be specifically identified by number or title and the MDL for each analyte expressed in the appropriate method reporting units. If the analytical method permits options which affect the method detection limit, these conditions must be specified with the MDL value. The sample matrix used to

determine the MDL must also be identified with the MDL value. Report the mean analyte level with the MDL. If a laboratory standard or a sample that contained a known amount analyte was used for this determination, report the mean recovery, and indicate if the MDL determination was iterated.

If the level of the analyte in the sample matrix exceeds 10 times the MDL of the analyte in reagent water, do not report a value for the MDL.

### Reference

Glaser, J. A., Foerst, D. L., McKee, G. D., Quave, S. A., and Budde, W. L., "Trace Analysis for Wastewaters," *Environmental Science and Technology*, 15, 1426 (1981).

*Table of Students' t Values at the 99 Percent Confidence Level*

<i>Number of Replicates</i>	<i>Degrees of Freedom (n-1)</i>	<i>t<sub>(n-1, 1-α = .99)</sub></i>
7	6	3.143
8	7	2.998
9	8	2.896
10	9	2.821
11	10	2.764
16	15	2.602
21	20	2.528
26	25	2.485
31	30	2.457
61	60	2.390
∞	∞	2.326

**APPENDIX B**

**LABORATORY QUALITY ASSURANCE PLAN  
BROWN AND CALDWELL LABORATORIES**

KLEINFELDER, PROJECT 50-1014-3

Prepared by: Brown and Caldwell Laboratories

Contact Person: Bob Peak, telephone number 818/795-7553  
Quality Assurance Director

This plan is modeled after guidelines presented in Test  
Methods for Evaluating Solid Waste, Third Edition, September  
1986, USEPA

*Audrey L. Morris - Sec'y for*  
Jane Freemyer  
Client Services Manager



## TABLE OF CONTENTS

1. Title and Signature Page
2. Table of Contents
3. Project Description
4. Project Organization
5. QA Objectives
6. Sampling Procedures
7. Sample Custody
8. Calibration Procedures and Frequency
9. Analytical Procedures
10. Data Reduction, Validation, Reporting
11. Internal Quality Control Checks
12. Performance/System Audits
13. Preventive Maintenance
14. Specific Routine Procedures Used to Assess Data,  
Precision, Accuracy, Completeness
15. Corrective Action
16. QA Report to Management

### 3. PROJECT DESCRIPTION

The project description is discussed in the main text presented by J.H. Kleinfelder and Associates.

### 4. PROJECT ORGANIZATION

Jane Freemyer will be the Brown and Caldwell Project Manager for this project. She will be responsible for insuring that the work is processed properly within Brown and Caldwell. She will maintain the primary contact with the client.

Paul Duerksen will be the Quality Assurance Coordinator. He will be responsible for all questions concerning Quality Assurance on this project.

### 5. QUALITY ASSURANCE OBJECTIVES

Our quality assurance program is designed to ensure the precision and accuracy of all analytical results. Control of this objective is maintained by adherence to specified operating procedures, use of quality control samples and standards, and observance of sample custody requirements.

The Quality Control acceptance limits for Methods 8010 and 8020 are presented in Tables 1.10-6 and 1.10-7. Both are attached.

### 6. SAMPLING PROCEDURES

The sampling procedures are outlined in the main text presented by J.H. Kleinfelder and Associates.

### 7. SAMPLE CUSTODY

Chain-of-custody procedures have been established to document the identity of a sample and its handling throughout the project. The sampling technician in the field initiates a chain-of-custody record. The chain-of-custody will be supplied by J.H. Kleinfelder and Associates.

Each sample is assigned a discrete log number which, in addition to being attached to the sample container, is entered on the custody record, in the legally required sample log book, and onto the computerized data handling system. Besides the log number, the computerized record also contains the client name, the sample description, the sample matrix type, the required analytical parameters, and the report due date. Supplementary information such as special handling requirements may be entered as well.

Verification of sample integrity is one of the main responsibilities of the sample control staff. The sample is inspected to see that:

1. The sample is clearly marked and dated.
2. The sample was collected in an appropriate container.
3. The sample is properly preserved.
4. There is sufficient volume to do all the analyses required.
5. The sample is received in good condition and the custody seal (if used) is intact.
6. Chain-of-custody forms match the number and description of samples.

If the above conditions are met, the sample will be given a log number and the relevant information recorded. If aliquots or subsamples are to be split out, care is taken to ensure that the subsamples are representative of the original. Blending or grinding may be required.

The sample control staff distributes the sample (or fractions of it, if the sample requires subdivision) into designated storage areas. Most samples are stored under refrigeration at four degrees C; refrigerators are marked with test categories for convenient retrieval of samples. Volatile organic vials are segregated from other samples to prevent vapor-phase cross contamination.

## 8. CALIBRATION PROCEDURES AND FREQUENCY

### A. Standard Curves

Spectrophotometers are standardized daily with at least three concentrations for every test. Absorbances are recorded in the bound notebook associated with each test; this step permits detection of any change in absolute performance. Indications of declining sensitivity or wavelength inaccuracy are corrected by having the unit professionally serviced.

Gas chromatograph and GC/MS determinations are standardized with curves containing at least three points as described in the October 26, 1984 Federal Register. The curves are verified on a daily basis with a quality control standard.

Standard solutions are prepared at intervals specified in the referenced methods. Where no limit is stated, they are replaced at intervals short enough to prevent detectable deterioration during the lifetime of the material. Even when no deterioration is detected, standards are replaced at least once every six months. Standards are obtained from suppliers using NBS traceable materials.

### B. Internal Standards and Surrogates

Internal standards are added to concentrated sample extracts of semivolatile priority pollutants and to the purging aliquots of volatile priority pollutant samples. Internal Standards serve to detect losses during capillary column injection of semivolatiles or during the purging volatile priority pollutants. They also provide a means for applying corrections to individual sample results, providing that such losses are not extraordinarily large.

Surrogates are typically spiked into the samples prior to extraction and thereby provide recovery data for sample workup. The recovery data can be used to identify systematic recovery problems or sample-specific extraction problems.

## 9. ANALYTICAL PROCEDURES

### A. Sources

Brown and Caldwell Laboratories makes extensive use of methods prescribed by the USEPA. Other methods are taken from Standard Methods for the Examination of Water and Wastewater, 16th Edition, APHA-AWWA-WPCF, 1985.

Primary USEPA sources of methods for the analysis of aqueous samples include:

Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, EPA 600/4-82-057, July 1982.

Methods for the Chemical Analysis of Water and Wastes, EPA 600/4-79-020, Revised, March 1983.

Federal Register, 40 CFR Part 136, October 26, 1984.

Soil and other solid samples are analyzed according to the procedures of Test Methods for Evaluating Solid Waste, USEPA SW-846, July 1982.

A number of additional EPA methods are summarized in the AB 1803 Methods Manual issued by the California Department of Health Services, 1984.

Air analysis is based on the NIOSH Manual of Analytical Methods, 2nd and 3rd editions, issued by the National Institute of Occupational Safety and Health of the Public Health Service.

Additional methods for various sample types are taken from such sources as the United States Geological Survey (USGS), The American Society for Testing and Materials (ASTM), and The Association of Official Analytical Chemists (AOAC).

Finally, a number of special methods have been developed by Brown and Caldwell Laboratories or adapted from literature sources for specialized applications. These methods are written up and maintained in Brown and Caldwell Laboratories methods reference file.

B. Examples

A listing of methods used by Brown and Caldwell Laboratories priority pollutant analysis in aqueous samples and solid wastes is given in below. Also included are methods for hazardous waste analysis.

METHODS FOR PRIORITY POLLUTANT AND HAZARDOUS WASTE ANALYSIS

<u>Determination</u>	<u>EPA Method Number</u>
Volatile organics by GC/MS	8240
Semivolatile organics by GC/MS	8270
Organochlorine pesticides and PCB's by GC/ECD	8080
Cyanide	9010
Phenolics, total	9065
Antimony	7041 or 6010
Arsenic	7060
Beryllium	7091 or 6010
Cadmium	7031 or 6010
Chromium	7191 or 6010
Hexavalent Chromium	7196
Copper	7211 or 6010
Lead	7421 or 6010
Mercury	7471
Nickel	7520 or 6010
Selenium	7740
Silver	7760 or 6010
Thallium	7841 or 6010
Zinc	7950 or 6010
Ignitability	1010
EP Toxicity	1310
Chlorophenoxy Herbicides	8150

Note: GC/MS is Gas Chromatography/Mass Spectrometry and GC/ECD is Gas Chromatography with Electron Capture Detection.

## 10. DATA REDUCTION, VALIDATION AND REPORTING

### A. Computer Management

Brown and Caldwell Laboratories utilizes a computerized sample control and data management system for collecting and reporting analytical data. Upon receipt, a sample is logged into the computer to initiate the process. Associated with each sample is a unique log number, client sample description, sample matrix type, the required analysis, and due date. Also included is supplementary information such as special handling requirements.

Each evening the data input operator enters the day's results into the computer, where they are transferred to "work awaiting approval" (WAA) status. The next morning the computer prints the previously entered results on work approval sheets, which each analyst or designated group leader checks against the original data as recorded in the analytical notebook for that parameter. The reviewer initials the entry to signify approval or makes corrections if necessary.

Later that day the initials are entered into the computer, which then transfers the data to the "report" file. When all the results for a particular sample have been entered and approved, a final report is printed. The printed report is reviewed by the section supervisors and signed by the Laboratory Manager before mailing to the client.

This computerized system maintains a complete audit trail for the work done on each sample. Information such as date sampled, date received, analyst's name, date completed, and the analytical method used in each determination are all retrievable from the data base.

## B. Archiving

Following analysis, all samples are kept for a minimum of 30 days. In this way, questions raised during the review of data can be addressed by inspection of the sample or by complete reanalysis using a different method. Analysis for components subsequently added to the list of parameters to be measured and incompatible with previous sample workup may be analyzed without resampling. The chief quality assurance feature provided by the archived samples is the ability to use the archived samples to resolve problems which may be noted only as the data are compared and interpreted.

Raw data for most procedures are kept in bound notebooks associated with the test or group of determinations. The completed notebooks are filed in the laboratory for ready reference should future comparisons be desirable. Raw chromatographic data, such as that collected in the quantitation of trace organics, are filed to permit critical reexamination at any time. Such information is particularly useful in cases where the inspection of old data may yield clues regarding the presence of newly identified species.

Raw GC/MS chromatograms are similarly archived together with the spectra of chromatographically isolated but unidentified components. In addition, raw GC/MS data are transferred to tape in an EPA-approved format in order to permit reprocessing to retroactively search for new classes of compounds or otherwise reexamine previously reported findings.

In addition to computer storage, a hard-copy printout of every sample report is filed under the client's name and saved for five years.

## C. Detection Limits

The method detection limit is often defined as the minimum concentration of analyte which can be identified, measured and reported with 99 percent confidence that the concentration is greater than zero. The method detection limit is defined as three times the standard deviation of seven replicate analyses of the analyte in question. The seven replicate determinations must be nonconsecutive, and the analyte concentration must be within five times the estimated detection limit.

When a sample is diluted, the reported detection limit is equivalent to the method detection limit multiplied by the dilution factor. The procedure for establishing the method detection limit is provided in Appendix B of 40 CFR Part 136 as published in the Federal Register of October 26, 1984.



#### D. Assessment

Before the significance of analytical data can be assessed, one needs to know how precise, how accurate, and how complete are the data subsets. Precision is amenable to strict definition by the analysis of replicate results according to schemes outlined in the USEPA Handbook for Analytical Quality Control in Water and Wastewater Laboratories, March, 1979, Chapter 6. Accuracy is somewhat more difficult to assess. Spike recovery determinations, regular analysis of laboratory control standards, and use of external check samples contribute to the general assurance that the accuracy of a determination is within acceptable limits. The ultimate accuracy of a determination also depends on factors external to the laboratory, such as sampling and storage conditions. Completeness is the most difficult characteristic to assess, depending as it does upon such factors as representativeness of sampling and subsampling, selection of the appropriate analytical parameters, and scope of the sampling program relative to the size of the environmental question being addressed.

While ultimate assessment depends on the experienced judgment of knowledgeable individuals, statistical treatment of the data can provide some objective measure of their soundness. The Federal Register of October 26, 1984, includes required calculations for accuracy on spiked samples for several organics methods. The same calculation may be used for any test amenable to spiking:

$$P = 100 (A - B)/T$$

Where

P = Percent spike recovery

A = Concentration determined on spiked sample

B = Concentration determined on original unspiked sample

T = True value of spike added

Precision values may be calculated from analysis of duplicate pairs. In his manual, Quality Assurance of Chemical Measurements, John K. Taylor of the National Bureau of Standards provides a formula for calculating the standard deviation based on a series of duplicates. When a sufficient number of spiked samples or duplicate pairs (at least 20) have been analyzed, control charts may be calculated. Because of the wide diversity of sample types and many different determinations, control charts have usually been calculated only in connection with special programs requiring them. However, the laboratory computer system is now being programmed to calculate and maintain charts for every test on every type of sample. For accuracy, the mean recovery and standard deviation ( $\bar{S}$ ) are calculated. Warning limits are set at  $\pm 2S$  and control limits at  $\pm 3S$ .

## 11. INTERNAL QUALITY CONTROL CHECKS

### A. Blanks

#### 1. Method Blanks

Method blanks are introduced on a daily basis or at a frequency of one-in-twenty if more than that number of determinations are run in one day. The blanks consist of organic-free or deionized water, which is then carried through the analytical scheme as for real samples. They serve to locate any contamination associated with laboratory storage, laboratory instrumentation, or equipment coming in contact with the sample during processing. They provide the fundamental baseline against which the sample signal is measured.

#### 2. Field and Travel Blanks

These closely related types of blanks find their most extensive application in analysis for volatile organic compounds. For each kind, the blank begins as organic-free reagent water in the laboratory. For a travel blank, a sample vial is filled with the reagent water at the laboratory and then carried with the sample containers out to the field and back to the laboratory. Field blanks are carried to the sample site in a separate container, with a vial being filled at the sampling location. In either case, the blank serves to identify the possibly correct for contamination associated with collecting or transporting the sample.

#### 3. Sample Blanks

These blanks come into play when native sample characteristics--most often color or turbidity--interfere with a determination. If the method in question relies on spectrophotometric detection, the sample's original absorbance at the wavelength of interest is measured and subtracted from the absorbance of the final developed color. The value is recorded in the bound bench book so both the apparent concentration and the corrected value may be calculated.

D. Duplicates and Spikes

Every tenth sample is analyzed in duplicate. The duplicate aliquots are carried through the entire workup and analytical scheme. The automated log-in system on the computer assigns samples for duplication based on designated matrix type. This assures that samples such as soils and hazardous wastes are duplicated at least as frequently as waters and wastewaters. How closely the results compare with each other provides a measure of precision for the determination.

If sufficient quantity exists, the same sample that is duplicated is also subjected to a spike analysis. In this technique, a known quantity of the analyte is added to a third aliquot of the sample. The recovery on the resulting spiked sample relative to its theoretical value reflects the accuracy of the determination. Since percent recoveries are often strongly influenced by the sample matrix, spiking real samples provides information on interferences as well as method performance.

Duplicate results and spike recoveries are sorted by matrix type for statistical analysis and calculation of control charts. If a particular determination is not carried out frequently enough on a particular matrix type for successful statistical manipulation, the results are grouped with others of similar matrices.

E. Laboratory Control Standards

These standards are purchased or prepared independently of method calibration standards. For every ten samples logged in for a particular determination, the computer assigns a laboratory control standard (LCS). The true value and the recovered concentration are archived along with duplicate and spike results.

## 12. PERFORMANCE/SYSTEMS AUDITS

### A. Laboratory Control Standards and Check Samples

Certified reference materials are acquired from the National Bureau of Standards for metals in tissue and sediment-like matrices. USEPA provides performance evaluation standards for both inorganics and organics. Additional certified reference materials are provided by the American Industrial Hygiene Association as part of its laboratory certification program. Using external sources for control standards ensures uniform and accurate results.

### B. Certification Programs

The laboratories are subjected to performance audits initiated several times every year. Recent audits have included:

1. USEPA semiannual drinking water performance check samples (WS series).
2. USEPA semiannual wastewater performance check samples (WP series).
3. California Department of Health Services (DOHS) certification for the analysis of priority pollutants and agricultural pesticides in well water.
4. Arizona Department of Health Services certification for general mineral analysis and fumigants (EDB, DBCP) in drinking water.
5. Orange County Environmental Management Agency approval to analyze for metals, inorganics, halogenated pesticides and PCBs.
6. California DOHS certification for complete chemical analysis of hazardous wastes.
7. American Association for Laboratory Accreditation.

In addition, a number of clients maintain laboratory certification programs that include performance check samples and on-site audits of facilities and procedures.

C. Brown and Caldwell Interlaboratory Comparisons

The exchange of samples between laboratories is a widely recognized quality assurance measure that provides information about procedural errors, contamination unique to a particular laboratory, and interlaboratory precision and accuracy. Because Brown and Caldwell Laboratories consists of two independently operated but similarly equipped laboratories, interlaboratory studies may be carried out with particular convenience. In this way one laboratory can serve as the external quality assurance unit for the other. The sample control staff of each laboratory is responsible for splitting samples for such comparative purposes. The interlaboratory exchange program is usually reserved for major projects with special QA/QC requirements.

D. Round Robin Studies

Brown and Caldwell Laboratories frequently participates in studies of methods or performance among groups of well-qualified environmental laboratories. Samples for such studies have been provided by Los Angeles County Sanitation Districts, Orange County Sanitation Districts, Electric Power Research Institute, and other public agencies and trade associations.

E. Audits

Each lab is inspected annually by the State of California, in support of the hazardous waste and drinking water certifications, as well as other state and county certifications.

In addition to external audits addressed in Section B, the Quality Assurance Director audits each lab on a semi-annual basis.

### 13. PREVENTIVE MAINTENANCE

Routine equipment maintenance and instrument service is performed on an appropriate service schedule basis. Major instruments such as balances, gas chromatographs, atomic absorption spectrophotometers, and the GC/MS systems are maintained under commercial service contracts or by in-house service technicians. Calibration, sensitivity, and response checks on a daily basis establish unscheduled service needs for analytical instruments.

14. SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA  
PRECISION, ACCURACY, AND COMPLETENESS

A. Organics by Gas Chromatography

Specialized quality control procedures for these analyses are typified by those used for pesticides and PCBs by EPA method 608. For every nine samples analyzed, another three QC samples--a method blank, a duplicate and a spike--are run. One QC control sample is analyzed for every ten client samples. The results are compared to the acceptance criteria listed by EPA in the Federal Register. If the result for a particular parameter does not meet the criteria, the sample is reanalyzed for that parameter. The instrumentation used in the analysis of aqueous samples include Hewlett-Packard 5890 Gas Chromatographs as well as Varian 3400 Gas Chromatographs fitted with Tekmar Purge and Trap sample injection systems. Both types of Gas Chromatographs use a Hall detector for 601 analysis and Photoionization Detector (PID) for 602 analysis.

B. Organics by GC/MS

1. Volatile Organics; EPA Method 624

Prior to the analysis of samples, and after meeting the calibration criteria for 50 ng Bromofluorobenzene (BFB), the GC/MS system is initially calibrated at a minimum of five concentrations to determine the linearity of response. Linear calibration curves and response factors for each of the EPA Method 624 compounds are periodically established by analyzing five concentrations over a 5-to-200 ug/L range. These response factors are verified every 12 hours by analyzing a QC check sample. Daily operation also includes tuning the GC/MS system with bromofluorobenzene (BFB) to meet instrument performance criteria established by the EPA. For every nine samples analyzed, a method blank, a duplicate, and a spike are also run. Three surrogates and three internal standards are added to each sample in order to monitor purging efficiency and instrument operation.

2. Base/Neutral and Acid Extractable Organics; EPA Method 625

Before any samples are analyzed, the GC/MS system must be tuned to meet the ion abundance criteria for a 50 ng Decafluorotriphenylphosphine (DFTPP) sample every 12 hours. Five-point calibration curves for each of the EPA Method 625 parameters are established periodically. The corresponding response factors are verified every 12 hours by analyzing a QC check sample. Daily ion abundance criteria are met by tuning the instrument against a decafluorotriphenylphosphine (DFTPP) standard.



Five surrogate standards are added prior to extraction of the sample in order to monitor the extraction efficiency of the method. A daily sensitivity check is done by adding six internal standards in each sample extract. In addition, three quality control samples--a method blank, a duplicate and a spike--are analyzed every batch of nine samples.

#### C. Metals Analysis

Most metals analyses are done by one of three techniques: flame atomic absorption spectrophotometry (FAA), graphite furnace atomic absorption spectrophotometry (GFAA), or inductively coupled argon plasma emission spectrophotometry (ICP). Available supplementary techniques include cold vapor and hydride generation. All sample digestions follow EPA or Standard Methods prescribed procedures. A daily method blank is run for each element. A three-point calibration curve is determined daily for each element. Calibration standards for that curve are prepared by dilution of 1000 mg/L certified standards obtained from commercial sources.

When the concentration of the metal being determined exceeds the highest standard, the sample is diluted so it falls within the range of calibration. A daily laboratory control standard is run for all metals. The accuracy of analysis of metals in soils is checked periodically by analyzing a National Bureau of Standards reference material such as SRM #1646, an estuarine sediment.

#### D. Selected General Chemistry Procedures

##### 1. Biochemical Oxygen Demand (BOD)

Samples for five-day BOD are stored at 4 degrees C and set within 48 hours of receipt. To test the quality of the dilution water, its dissolved oxygen is measured initially and at the end of the five-day incubation period. Three dilutions of each sample are set. The oxygen depletion of at least one of the dilution must be at least two mg/L; the final dissolved oxygen content must be greater than one mg/L. One duplicate and one glucose-glutamic acid laboratory control standard are set with each batch of samples.

## 2. General Mineral Analysis

Several checks on the accuracy of the analysis of drinking water for general minerals are applied. The anion cation balance must agree to within +/- 5%. Also, the total dissolved solids content should fall within 65 to 75% of the specific conductance.

## 3. Colorimetric Analysis

Chemical oxygen demand, cyanide, phenol, nitrate, nitrite, and phosphate can all be done colorimetrically. Each analysis requires a three-point calibration. A linear regression is performed to ensure that operating conditions are in order and that the analyst is working in the linear range. Typical values for the correlation coefficient exceed 0.995.

## 4. Titrimetric Analyses

Hardness, alkalinity, chloride, free CO<sub>2</sub>, and chemical oxygen demand can all be determined titrimetrically. Titrants are standardized using primary standards.

## 5. Gravimetric Analyses

Oil and grease, total solids, dissolved solids, suspended solids and gravimetric sulfates all fall in this category. Each analysis depends heavily on the accuracy of the balance used. For this reason, balances are checked each week against class "S" weights. Desiccants are monitored to ensure a "moisture free" environment during storage of samples. Oven temperatures are also monitored regularly to ensure compliance with those specified in the EPA Methods.

E. Microbiology

Certification requirements of the California Department of Health Services control the principal features of microbiology QC. These features include daily recording of all incubator temperatures, recording and filing of the autoclave performance record, sterilization of sample containers, application of a dechlorinating agent to sample containers, and monthly performance of a completed coliform test to verify routine confirmed coliform results.

An annual water suitability test is run to make sure the purified water used in media preparation contains no growth-promoting or inhibitory substances. An inhibitory residue test is carried out annually on glassware to verify that routine cleaning procedures will not adversely affect results.

F. Fish Bioassay

Fish toxicity bioassays are carried out according to California Department of Fish and Game guidelines, using specified test organisms, sample dilutions, and sample volumes. They are conducted in a constant-temperature room in which fish are acclimated for seven days prior to use in tests. Checks performed before and during tests on the control tank and on all dilutions are pH, temperature, and dissolved oxygen level. Freshwater bioassays are checked for alkalinity and hardness while saline or brackish bioassays are checked for conductivity at the beginning of the test. A test is considered invalid if there is more than 10% mortality among control fish during the test. Temperatures of test solutions must be within the specified test range for the organism used. Confidence limits are established using procedures provided by California Department of Fish and Game.

## 15. CORRECTIVE ACTION

The control charge, predetermined acceptance limit, or EPA acceptance criteria serve as alert systems for unsatisfactory or unexpected results. The nature of corrective action may take several forms, but the first step is usually to repeat the analysis on the sample which failed. If the repeat does not replicate the failure, and prior and subsequent QC data do not indicate a systematic error, the value may be treated as a random error and disregarded.

More commonly, diagnosis and correction of an analytical problem will follow. If the repeat analysis continues to show difficulty, the analyst will bring it to the section supervisor's attention. If the required correction is not readily apparent, the supervisor will call in the laboratory technical director and the quality assurance coordinator. Together, they plan a series of steps to isolate and correct the problem. Based on frequency of occurrence and correctability, the usual checking order includes:

1. Check the calculations.
2. Check lab control standard (this may reveal systematic errors).
3. Examine the sample for non-homogeneity or peculiar interferences.
4. Check instruments for proper performance.
5. Verify that standard solutions are fresh and properly prepared.
6. Assure the purity of reagent water or reagent gases.
7. Closely observe the analyst to be certain no procedural errors are occurring.

During the troubleshooting process, routine analysis for that determination is discontinued. Once the problem is found and corrected, the best estimate is made of when the problem first occurred. Data collected after this critical point is discarded. If possible, all analyses since the last valid control check will be repeated. Analyses performed after the resolution of the problem must be accompanied by more duplicates and spikes than the regular ten-percent level. The higher level of QC continues until the section supervisor and the quality assurance coordinator are satisfied that the problem has been completely solved.

To assist efficient resolution of problems, the QA coordinator maintains a test-based file of previous corrective actions. The material is readily available to both laboratories. Since some determinations may be subject to common errors, this program helps reduce the time required to correct a problem if the other Brown and Caldwell Laboratory has previously dealt with the same issue.

#### 16. QUALITY ASSURANCE REPORT TO MANAGEMENT

A formal quality assurance report will be presented upon request. The format will be as presented in the previous text.

**BROWN AND CALDWELL LABORATORIES**

373 SOUTH FAIR OAKS AVENUE PASADENA, CA 91105 • (818) 795-7553

**BROWN AND CALDWELL LABORATORIES****ACCEPTANCE LIMITS****Aqueous Samples**

Parameter	Warning Limits (%)			Control Limits (%)		
Bromodichloromethane	64	-	150	42	-	172
Bromoform	37	-	135	13	-	159
Bromomethane	24	-	120	D	-	144
Carbon tetrachloride	60	-	126	38	-	143
Chlorobenzene	57	-	131	38	-	150
Chloroethane	61	-	122	46	-	137
2-chloroethylvinyl ether	43	-	157	14	-	186
Chloroform	63	-	119	49	-	133
Chloromethane	32	-	161	D	-	193
Dibromochloromethane	52	-	163	24	-	191
1,2-Dichlorobenzene	35	-	173	D	-	208
1,3-Dichlorobenzene	37	-	157	7	-	187
1,4-Dichlorobenzene	59	-	126	42	-	143
1,1-Dichloroethane	61	-	118	47	-	132
1,2-Dichloroethane	67	-	131	51	-	147
1,1-Dichloroethene	51	-	144	28	-	167
trans-1,2-Dichloroethene	58	-	136	38	-	155
1,2-Dichloropropane	63	-	137	44	-	156
cis-1,3-Dichloropropene	48	-	152	22	-	178
trans-1,3-Dichloropropene	48	-	152	22	-	178

**BROWN AND CALDWELL LABORATORIES**

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**BROWN AND CALDWELL LABORATORIES****Acceptance Limits****Method 8010: Halogenated Volatile Organics**

Parameter	Aqueous Samples			Control Limits (%)		
	Warning Limits (%)					
Methylene chloride	48	-	139	25	-	162
1,1,2,2-Tetrachloroethane	37	-	155	8	-	184
Tetrachloroethene	49	-	139	26	-	162
1,1,1-Trichloroethane	57	-	122	41	-	138
1,1,2-Trichloroethane	55	-	120	39	-	136
Trichloroethene	54	-	128	35	-	146
Trichlorofluoromethane	44	-	134	21	-	156
Vinyl chloride	50	-	140	28	-	163

**BROWN AND CALDWELL LABORATORIES**

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**BROWN AND CALDWELL LABORATORIES****Acceptance Limits****Method 8020: Aromatic Volatile Organics**

Parameter	Aqueous Samples		Control Limits (%)
	Warning Limits (%)		
Benzene	58	- 132	39 - 150
Chlorobenzene	68	- 122	55 - 135
1,2-Dichlorobenzene	56	- 134	37 - 154
1,3-Dichlorobenzene	65	- 126	50 - 141
1,4-Dichlorobenzene	59	- 126	42 - 143
Ethylbenzene	53	- 139	32 - 160
Toluene	63	- 131	46 - 148



Review Comments for the CP Chemicals, Inc. Closure Plan

1. The Sampling Plan does not address all the requirements of the corrective action order. As a minimum, the plan should include site background information; rationale for sample location and sample analyses; desired detection limits for all parameters; a sampling objective which describes the final uses of the data; and field and laboratory QA/QC measures. During revision of the Plan, Attachment 3 of the corrective action order may be used as a guide to contents of the Plan.
2. In Section 1.4, the following modifications are suggested.
  - a. The reasons why the parameters in Section 1.4 have been selected for analysis should be explained. What are the desired detection limits?
  - b. The analytical methods for metals, total alkalinity, and "ferrous iron" should be stated.
  - c. Methods 325.3, 350.3 and 340.1 have been designed for analysis of water samples. If these methods are to be used for soil samples, an extraction procedure must be specified. Method 9250 or 9251 is recommended for analysis of chloride.
  - d. The SW-846 Extraction Procedures Toxicity test specifies analytical parameters and a sample extraction procedure. If a modification to the E.P. Tox method is being proposed, this should be explained.
  - e. If analysis of soil samples for Ammonia is not desired, as stated in paragraph 1 of Section 1.4, it should be eliminated from the table.
3. Analytical holding times should be discussed in Section 1.6, and need to be considered prior to holding soil samples for future analysis. Soil samples to be analyzed for volatiles, semi-volatiles, PCBs and pesticides should be extracted in less than 14 days, and the extract should be analyzed within 40 days of extraction.
4. Collection of duplicate samples at a 10% frequency (or 1 per week, whichever is greater) is recommended for all parameters. The way in which the duplicates are collected should be described. The duplicate samples should be sent "blind" to the laboratory.
5. It is recommended that sample numbers, rather than sample point names, be written on sample labels. The sample names and corresponding sample numbers should be recorded in a bound field notebook.

6. Since the purpose of the sampling effort is to characterize the area surrounding pond #1, it is recommended that least 5 sample locations be selected on the accessible sides of the pond. Why aren't samples being taken on the east edge of pond #1? The rationale for sample location should be discussed. Also, why aren't background soil samples being taken at 1'?

7. A table summarizing the sampling locations, depths, and analytical requirements discussed in Section 1.3 would be helpful for both reviewers of the plan and for field sampling personnel.

8. Sample volumes and the parameters which will be combined in the brass sleeves should be stated. How will the brass sleeves be sealed? How will soil cuttings be disposed of?

9. How will the laboratory performing the soil analyses be selected? If review of the data is desired the lab must submit all raw data, including chromatograms, for samples, blank, spike, duplicate and instrument calibration runs.